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Final

Sampling and Analysis Plan Additional Investigations at Operable Unit 1, Site 17

Marine Corps Air Station Cherry Point
Cherry Point, North Carolina

NAVFAC
Naval Facilities Engineering Command
NAVFAC ATLANTIC

Prepared for

Department of the Navy
Naval Facilities Engineering Command
Atlantic Division

Contract No.
N62470-02-D-3052
CTO-0208

July 2008

Prepared by

CH2MHILL

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Contract Task Order 208

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Under the

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CH2MHILL

Virginia Beach, Virginia

SAP Worksheet #1—Title and Approval Page

Final
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
July 2008

Marine Corps Air Station Cherry Point, Additional Investigations at Operable Unit 1, Site 17

Prepared for:
Department of the Navy
Naval Facilities Engineering Command
Mid-Atlantic Division

Prepared by:
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Prepared under:
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CTO-208

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JEFF CHRISTOPHER / MCAS IRPM / DATE

Gena Townsend

GENA TOWNSEND / EPA RPM / DATE

George L Lane 8/15/08

GEORGE LANE / NCDENR RPM / DATE

Executive Summary

This Sampling and Analysis Plan (SAP) is prepared to support the proposed field activities at Operable Unit 1 (OU1) Site 17 at Marine Corps Air Station (MCAS) Cherry Point, North Carolina. This United States Navy (Navy) specific SAP includes 37 worksheets that detail various aspects of the environmental investigation process and serves as a guideline for the field activities and data quality assessment. This SAP was developed in accordance with two guidance documents: 1) U.S. Environmental Protection Agency (USEPA), *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS* (USEPA, 2002), and 2) USEPA, *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP) (USEPA, 2005). The Data Quality Objectives (DQOs) were prepared using USEPA's seven-step DQO process.

The Navy, Naval Facilities Engineering Command (NAVFAC), Mid-Atlantic Division, is conducting the Site Investigations under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA). USEPA Region 4 is the lead regulatory agency.

The Site Investigation will help to characterize the occurrence of dieldrin and polychlorinated biphenyls (PCBs) at Site 17, within soil and groundwater. At Site 17, one surface soil and one groundwater sample will each be collected at 10 separate locations for PCBs and 6 separate locations for dieldrin. The site investigations will be conducted in one field mobilization.

This SAP will help ensure that environmental data collected or compiled are scientifically sound, of known and documented quality, and suitable for intended uses. The laboratory information cited in this SAP is for the contracted laboratories that provide analytical services for this investigation. The analytical services for this investigation will be provided by GPL Laboratories. Data validation services will be provided by DataQual Environmental Services.

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2 Site Location Map—Site 17
3 Proposed Sampling Locations at Site 17
4 Simplified Conceptual Site Model of Site 17
5 Decision Tree—Site 17

Appendixes (provided on CD-ROM)

A Laboratory SOPs
B Field SOPs
C Data Management Documents

Abbreviations and Acronyms

AA	Atomic Absorption
ANSI/ASQ	American National Standards Institute/American Society for Quality
ASTM	American Society for Standards and Materials
BOD	Biological Oxygen Demand
CA	Corrective Action
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CLP	Contract Laboratory Program
COC	Contaminant of Concern
CRDL	Contract-Required Detection Limit
CSM	Conceptual Site Model
CTO	Contract Task Order
CWA	Clean Water Act
DCN	Document Control Number
DoD	Department of Defense
DQI	Data Quality Indicator
DQO	Data Quality Objective
EPA	Environmental Protection Agency
FCR	Field Change Request
FS	Feasibility Study
FSP	Field Sampling Plan
GC	Gas Chromatograph
GC/MS	Gas Chromatograph/Mass Spectrometer
GIS	Geographic Information System
GPC	Gel Permeation Chromatography
GPS	Global Positioning System
GW	Ground Water
ICP	Inductively Coupled Plasma
IDQTF	Intergovernmental Data Quality Task Force
LCS	Laboratory Control Sample
LFB	Laboratory Fortified Blank
LIMS	Laboratory Information Management Systems
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols (Manual)
MARSSIM	Multi-Agency Radiation Survey and Site Investigation Manual
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MOU	Memorandum of Understanding

MPC	Measurement Performance Criteria
MQO	Measurement Quality Objectives
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MSR	Management Systems Review
NEIC	National Enforcement Investigations Center
NIST	National Institute of Standards and Technology
NPDES	National Pollutant Discharge Elimination System
NPL	National Priorities List
PA/SI	Preliminary Assessment/Site Investigation
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PCB	Polychlorinated Biphenyl
PDF	Portable Document Format
PG	Professional Geologist
PM	Project Manager
PQO	Project Quality Objective
PRP	Potentially Responsible Party
PRQL	Project-Required Quantitation Limit
PT	Proficiency Testing (previously known as performance evaluation (PE) sample)
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
QMP	Quality Management Plan
QS	Quality System
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
RSL	Regional Screening Level
RT	Retention Time
RTM	Remedial Technical Manager
SAP	Sampling and Analysis Plan
SD	Standard Deviation
SDG	Sample Delivery Group
SDWA	Safe Drinking Water Act
SNEDD	Supplemental Naval Installation Restoration Information Solution Electronic Data Deliverable
SOP	Standard Operating Procedure
SQL	Sample Quantitation Limit

SRM	Standard Reference Material
SVOA	Semivolatile Organic Analytes
SVOC	Semivolatile Organic Compounds
SW	Surface Water
TBD	To Be Determined
TCLP	Toxicity Characteristic Leaching Procedure
TSA	Technical Systems Audit
UFP	Uniform Federal Policy
USACE	United States Army Corps of Engineers
VOA	Volatile Organic Analytes
VOC	Volatile Organic Compounds
VSP	Visual Sample Plan

SAP Worksheet #2—SAP Identifying Information

Site Name/Number: Site 17
Operable Unit: OU1
Contractor Name: CH2M HILL
Contract Number: N62470-02-D-3052 CTO-208
Contract Title: Navy Clean III

1. This SAP was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (U.S. EPA 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (U.S. EPA 2002)*.

2. Identify regulatory program: CERCLA

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Partnering Meeting	October 2006
Partnering Meeting	July 2007
Meeting between Bill Hannah and Doug Bitterman	October 15, 2007
Communication between Bill Hannah and Dan Lavoie	October 24, 2007
Partnering Meeting	December 4, 2007
Partnering Meeting	February 6, 2008
Partnering Meeting	March 18, 2008

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
IT Corporation, 1994. *Final Construction Sampling and Analysis Plan, Sites 5 and 17, US Marine Corps Air Station, Cherry Point, North Carolina.*	November, 1994

6. List organizational partners (stakeholders) and connection with lead organization:

Lead Organization: U.S. Navy (NAVFAC, Mid-Atlantic); Lead Regulatory Agency: USEPA Region 4; State Regulatory Agency: North Carolina Department of Environment and Natural Resources (NCDENR).

7. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

All required SAP elements are provided in this document. The crosswalk table is not applicable and is removed from this document.

SAP Worksheet #3—Distribution List

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
Janice Nielsen	Remedial Project Manager	NAVFAC Mid-Atlantic	Phone: (757) 322-8339 Fax: (757) 322-8280	Email: janice.nielsen@navy.mil (Mailing and FedEx address): Commander NAVFAC MIDLANT Attn: Janice Nielsen LRA, Building C, NC IPT 6506 Hampton Blvd Norfolk, VA 23508-1278	(A PANTAGON number will be assigned when the final document is being prepared.)
Jeff Christopher	Installation Restoration Program Manager	MCAS Cherry Point Environmental Affairs Department	Phone: (252) 466-4421 Fax: (252) 466-2000	Email: jeffrey.christopher@usmc.mil (Mailing address): MCAS Cherry Point PSC Box 8006 Cherry Point, NC 28533-0006 (FedEx address): MCAS Cherry Point Building 4223, Access Road Cherry Point, NC 28533-0006	(A PANTAGON number will be assigned when the final document is being prepared.)
Gena Townsend	Remedial Project Manager	USEPA Region 4	Phone: (404) 562-8538 Fax: (404) 562-8518	Email: townsend.gena@epa.gov (Mailing and FedEx address): USEPA Region 4 Atlanta Federal Center Waste Management Division Federal Facilities Branch 61 Forsyth St. SW Atlanta, GA 30303-3104	(A PANTAGON number will be assigned when the final document is being prepared.)
George Lane	Remedial Project Manager	NCDENR	Work Phone: (919) 508-8462 Emergency Phone: (336) 202-8665 Fax: (919) 733-4811	Email: george.lane@ncmail.net Home Email: GeorgeL100@aol.com (Mailing address): NC Department of Environmental and Natural Resources, Superfund Section PO Box 27687 Raleigh, NC 27611-7687 (FedEx address): NC Department of Environmental and Natural Resources, Superfund Section 401 Oberlin Rd., Suite 150 Raleigh, NC 27605	(A PANTAGON number will be assigned when the final document is being prepared.)
Bonnie Capito	Librarian	NAVFAC Atlantic	(757) 322-4785	bonnie.capito@navy.mil	(A PANTAGON number will be assigned when the final document is being prepared.)
Doug Bitterman	Activity Manager	CH2M HILL	(757) 671-6209 (703) 627-3291 (cell)	Doug.bitterman@ch2m.com	(A PANTAGON number will be assigned when the final document is being prepared.)

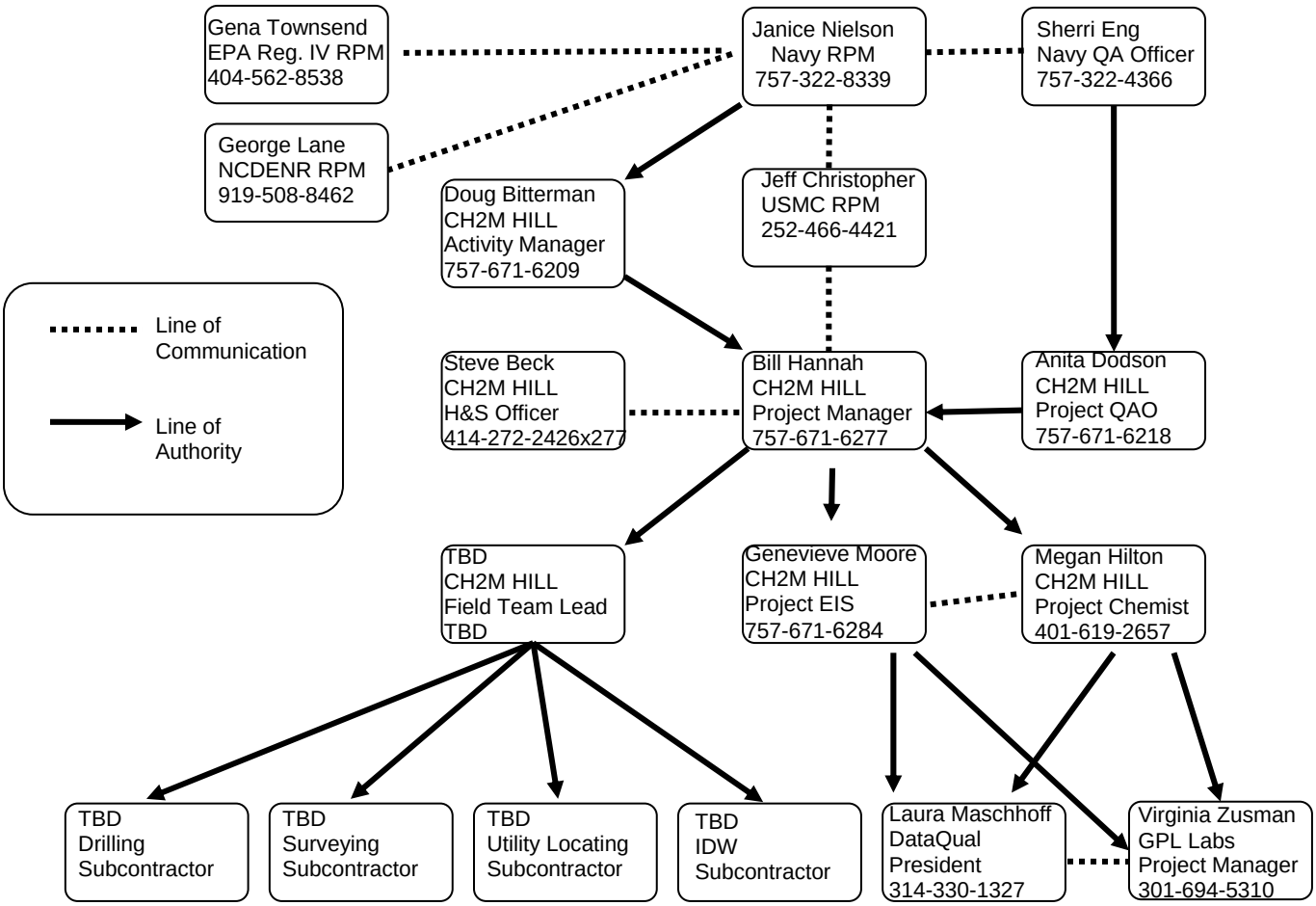
SAP Worksheet #3—Distribution List (continued)

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
Bill Hannah	Project Manager	CH2M HILL	(757) 671-6277	Bill.hannah@ch2m.com	(A PANTAGON number will be assigned when the final document is being prepared.)
Anita Dodson	Project QA Officer	CH2M HILL	(757) 671-6218	Anita.dodson@ch2m.com	(A PANTAGON number will be assigned when the final document is being prepared.)
Paul Favara	Program Quality Manager	CH2M HILL	(352) 335-5877 x52396	Paul.favara@ch2m.com	(A PANTAGON number will be assigned when the final document is being prepared.)
Megan Hilton	Project Chemist	CH2M HILL	(401) 619-2657	Megan.hilton@ch2m.com	(A PANTAGON number will be assigned when the final document is being prepared.)
Genevieve Moore	Project EIS	CH2M HILL	(757) 671-6284	Genevieve.moore@ch2m.com	(A PANTAGON number will be assigned when the final document is being prepared.)
TBD	Field Team Leader	CH2M HILL	TBD	TBD	(A PANTAGON number will be assigned when the final document is being prepared.)
TBD	Field Crew Members	CH2M HILL	TBD	TBD	(A PANTAGON number will be assigned when the final document is being prepared.)
Steve Beck	Health and Safety Officer	CH2M HILL	(414) 272-2426 x277	Steven.beck@ch2m.com	(A PANTAGON number will be assigned when the final document is being prepared.)
Roni Warren	Human Health Risk Assessor	CH2M HILL	(814) 364-2454	Roni.warren@ch2m.com	(A PANTAGON number will be assigned when the final document is being prepared.)
Bill Kappleman	Ecological Risk Assessor	CH2M HILL	(703) 376-5152	William.kappleman@ch2m.com	(A PANTAGON number will be assigned when the final document is being prepared.)
Virginia Zusman	Director of Project Management	GPL Laboratories	(301) 694-5310	Email: zusman@gplab.com Mailing address: 7210A Corporate Ct. Frederick, MD 21703	(A PANTAGON number will be assigned when the final document is being prepared.)
Laura Maschhoff	President	DataQual Environmental Services	(314) 330-1327	Email: dataqual@charter.net Mailing address: 5830 Amberway Drive St. Louis, MO 63128	(A PANTAGON number will be assigned when the final document is being prepared.)

SAP Worksheet #4—Project Personnel Sign-Off Sheet

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email Receipt	SAP Section Reviewed	Date SAP Read
Janice Nielsen	U.S. Navy/Remedial Project Manager	(757) 322-8339 (757) 617-0987 (cell)			
Doug Bitterman	CH2M HILL/Activity Manager and Activity Quality Manager	(757) 671-6209 (703) 627-3291 (cell)			
Jeff Christopher	MCAS Environmental Affairs Department/ Installation Restoration Program Manager	(252) 466-4421			
Gena Townsend	USEPA Region 4/ Remedial Project Manager	(404) 562-8538			
George Lane	NCDENR/ Remedial Project Manager	(919) 508-8462			
Bonnie Capito	NAVFAC Atlantic/ Librarian	(757) 322-4785			
Bill Hannah	CH2M HILL/Project Manager	(757) 671-6277			
Anita Dodson	CH2M HILL/Program Chemist	(757) 671-6218			
Paul Favara	CH2M HILL/Program Quality Manager	(757) 671-6209 (703) 627-3291 (cell)			
Megan Hilton	CH2M HILL/Project Chemist	(401) 619-2657			
Genevieve Moore	CH2M HILL/ Project EIS	(757) 671-6284			
Steve Beck	CH2M HILL/Health and Safety Officer	(414) 272-2426 x277			
TBD	CH2M HILL/Field Team Leader	TBD			
TBD	CH2M HILL /Field Crew Members	TBD			
Laura Maschhoff	DataQual/President	(314) 330-1327			
Virginia Zusman	GPL Labs/Director of Project Management	(301) 694-5310			

SAP Worksheet #5—Project Organizational Chart



SAP Worksheet #6—Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Point of Contact with Partnering Team	Navy RPM for MCAS Cherry Point	Janice Nielsen	(757) 322-8339	Primary point of contact for Navy; all materials and information pertaining to the project will be forwarded to the Partnering Team following review.
Environmental Manager	MCAS Cherry Point Installation Restoration Program Manager	Jeff Christopher	(252) 466-4421	Oversees all remedial activities at USMC Cherry Point. Any issues that may impact the Cherry Point operations are to be reported to him immediately.
Primary contact for CH2M HILL activities	CH2M HILL Activity Manager for MCAS Cherry Point	Doug Bitterman	(757) 671-6209	Primary point of contact for Navy and MCAS Cherry Point RPMs; oversees CH2M HILL project delivery for this project.
Manage all Project Phases	CH2M HILL Project Manager for this project	Bill Hannah	(757) 671-6277	Issues reported to the Navy RPM immediately and followed up in writing within 2 business days. Implement modifications to the SAP.
SAP changes in the field	CH2M HILL Field Team Leader (FTL)	TBD	TBD	Notify the PM by phone and email of changes to the SAP made in the field and the reasons within 24 hours. Changes will be documented.
Daily Field Progress Reports	CH2M HILL FTL	TBD	TBD	Field Team Leader will email or fax daily field progress reports to PM; telephone communication with project managers on as-needed basis.
Data tracking from collection through upload to database	CH2M HILL Environmental Information Specialist (EIS)	Genevieve Moore	(757) 671-6284	EIS will track data from sample collection through upload to database, ensuring SAP requirements are met by laboratory and field staff.
Reporting Lab Data Quality Issues	Laboratory Project Manager	Virginia Zusman GPL Labs	(301) 694-5310	All QA/QC issues with project filed samples will be reported by the lab to the EIS, Project Chemist, and Contractor Quality Assurance Officer within 2 business days.
Field and Analytical Corrective Actions	CH2M HILL Quality Assurance Officer	Anita Dodson	(757) 671-6218	The need for corrective action for field and analytical issues will be determined by the Field Team Leader and/or Contractor Quality Assurance Officer.
Release of Analytical Data	CH2M HILL Project Chemist	Megan Hilton	(401) 619-2657	No analytical data can be released until validation is completed and the Project Chemist has approved the release.

SAP Worksheet #7—Personnel Responsibilities and Qualifications Table

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Janice Nielsen	Remedial Project Manager	NAVFAC	Coordinates Environmental Restoration (CERCLA/MRP)activities at MCAS Cherry Point.	
Jeff Christopher	Installation Restoration Program Manager	USMC Cherry Point	Oversight of remedial activities at MCAS Cherry Point .	
Doug Bitterman	Activity Manager/Senior Consultant	CH2M HILL	Responsible for ERP at MCAS Cherry Point; Provides senior technical oversight and review.	B.S. Geology M.S. Civil Engineering 18 years experience
Bill Hannah	Project Manager	CH2M HILL	Directs and oversees staff and subcontractors. Develops SAP for Partnering Team and Navy review. Presents the findings of the investigation in a report for the Partnering Team for future site status decisions. Responsible for data usability evaluation and final decision-making.	B.S. Geology Professional Geologist: California 10 years experience
Anita Dodson	QA Officer	CH2M HILL	Responsible for audits, corrective action, check of QA performance; QA/QC of SAP.	B.S. Chemistry 14 yrs. experience
Megan Hilton	Project Chemist	CH2M HILL	Performs oversight of laboratory and data validators, releases analytical data, data usability evaluation.	B.S. Chemistry and Environmental Science 2 yrs. experience
TBD	Field Team Leader	CH2M HILL	Supervises field sampling and coordinates all field activities	
Steve Beck	Health and Safety Officer	CH2M HILL	Oversees health and safety for field activities	M.S., Occupational Safety and Health 13 yrs. experience
Genevieve Moore	Environmental Information Specialist (EIS)	CH2M HILL	Manages sample tracking, coordinates with laboratory and data-validator, data management	B.S. Biology .5 yr experience
Virginia Zusman	Laboratory Project Manager	GPL Laboratories	Manages analytical projects from initiation to completion.	
Laura Maschhoff	Data Validation Project Manager	DataQual Environmental Services	Responsible for contractual and administrative issues	
TBD	Driller	TBD	Operates equipment used to collect soil and groundwater samples.	

SAP Worksheet #8—Special Personnel Training Requirements Table

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates
Environmental Field Work	HAZWOPER 40 hour training and 8 hour refreshers	Various registered organizations	Annually	All field crew members	TBD/ CH2M HILL	CH2M HILL Human Resources Department
Environmental Field Work	3R Training (Recognize, Retreat, Report)	Internal, CH2M HILL	Project-specific	All field crew members	TBD/CH2M HILL	Document in personal HASP file
Site Safety Coordinator	Site Safety Coordinator-Hazardous Waste Training	Internal to CH2M HILL	Every 3 years	At least one field crew member must be designated as the SSC	SSC/ CH2M HILL	CH2M HILL Human Resources Department

SAP Worksheet #9-1—Project Scoping Session Participants Sheet

Project Name: Additional Investigations at OU1 Site 17 **Projected Date(s) of Sampling:** Summer 2008 **Project Manager:** Bill Hannah	**Site Name:** 17 **Site Location:** MCAS Cherry Point, North Carolina
Date of Session: October 12, 2006 **Scoping Session Purpose:** Partnering Team Meeting	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Jeff Christopher	Installation Restoration Project Manager	MCAS Environmental Affairs Department	Phone: (252) 466-4421	jeffrey.christopher@usmc.mil	Coordinates base environmental activities
Gena Townsend	Remedial Project Manager	USEPA Region 4	Phone: (404) 562-8538	townsend.gena@epa.gov	EPA regulator
George Lane	Remedial Project Manager	NCDENR	(919) 508-8462	george.lane@ncmail.net	State regulator
Rodger Jackson	Former Remedial Project Manager	NAVFAC Mid-Atlantic			No longer involved with project
Bill Friedmann	Former Activity Manager, MCAS Cherry Point	CH2M HILL			No longer involved with project
Katie Tippin	Engineer	CH2M HILL			No longer involved with project
Dan Lavoie	Biologist	CH2M HILL	(202) 290-1455	Daniel.Lavoie@CH2M.com	Conducted previous ecological assessments at Site 17

Comments/Decisions:

Rodger noted during the meeting that additional samples may need to be collected at Site 17. Gena suggested providing a work plan to collect samples.

Action Items:

N/A

Consensus Decisions:

N/A

SAP Worksheet #9-2—Project Scoping Session Participants Sheet

Project Name: Additional Investigations at OU1 Site 17 **Projected Date(s) of Sampling**: Summer 2008 **Project Manager:** Bill Hannah	**Site Name:** 17 **Site Location:** MCAS Cherry Point, North Carolina
Date of Session: July 11, 2007 **Scoping Session Purpose:** Partnering Meeting	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Janice Nielsen	Remedial Project Manager	NAVFAC Mid-Atlantic	(757) 322-8339	Email: janice.nielsen@navy.mil	Coordinates Environmental Restoration (CERCLA/MRP) activities
Rodger Jackson	Former Remedial Project Manager	NAVFAC Mid-Atlantic			No longer involved with project
Jeff Christopher	Installation Restoration Project Manager	MCAS Environmental Affairs Department	Phone: (252) 466-4421	jeffrey.christopher@usmc.mil	Coordinates base environmental activities
Gena Townsend	Remedial Project Manager	USEPA Region 4	Phone: (404) 562-8538	townsend.gena@epa.gov	EPA regulator
George Lane	Remedial Project Manager	NCDENR	(919) 508-8462	george.lane@ncmail.net	State regulator
Doug Bitterman	Activity Manager, MCAS Cherry Point	CH2M HILL	(757)-671-6209	Doug.Bitterman@CH2M.com	Overseeing UFP-SAP production, Work Plan production, and project support
Tim Wenk	Staff Consultant	CH2M HILL	(757)-671-6265	Tim.Wenk@CH2M.com	Project manager

Comments/Decisions:

Site 17

The site is where PCBs in transformer oil discharged into the DRMO drainage ditch in the 1960s. In 1995, a removal action was conducted to be protective of human health; the cleanup goal for the soil was 10 ppm. The RI indicated that there are currently no human health issues at the site and the risk is driven by ecological issues. The site had previously not been considered a viable habitat; however the Step 3A Addendum identified birds and upper trophic level receptors as being susceptible to site contaminants.

In a previous discussion during a site visit, Lynn Wellman, formerly with the EPA, had stated that a remedial goal of 1 ppm was appropriate for Site 17. Although the team remembers the level being stated, no one knew where this number came from and Rodger believes it was just a statement that Lynn made without any support or calculations behind it.

SAP Worksheet #9-2—Project Scoping Session Participants Sheet (continued)

Gena indicated that the contaminated media at Site 17 is soil more so than sediment; therefore, there is no aquatic habitat and a clean up level of 10 ppm is acceptable provided it does not leave an ecological risk at the site. To determine if the 10 ppm level is protective of ecological receptors, Gena suggested re-running the ecological risk assessment after removing the samples above 10 ppm to see if the resulting HQ is less than 1. The objective is to determine the action level at which a HQ slightly above 1 is achieved.

The RI shows 5 to 7 samples with PCBs>25 mg/kg, which is above human and ecological screening values. However, Rodger reiterated that the RI does not show HH risk and that the soil samples may have been considered sediment in the risk assessment. Based on the sample results, Gena and Rodger indicated there is a potential HH risk and, in order to get to a ROD, a HH risk assessment needs to be done. Doug said CH2M HILL would look into the level of effort to have a HH risk assessment re-done for Site 17 and will try to establish why the RI indicated there was not a HH risk associated with Site 17.

The RI states that groundwater doesn't appear to be impacted from Site 17; however, interpolated isoconcentration lines show otherwise. Gena indicated that is it necessary to collect additional samples to confirm pesticide/PCB concentrations in soil and groundwater. The goal of the sampling is to define the contamination area for pesticides/PCBs. For pesticides, Gena suggested sampling for dieldrin only, approximately 1 sample per 100 ft in the surface soil and to collect direct-push groundwater samples. The sampling scheme for PCBs (number of samples/locations) will have to be determined later during work planning activities.

Jan asked if soil samples are collected and the results are less than the NC Soil Screening Levels (SSLs), is it still necessary to collect groundwater samples just because we have seen contamination in the past. The team responded that no, that was not the case. Doug indicated that the detected concentrations for pesticides in groundwater may be false positives, as the laboratory method used for the analysis has shown to be problematic in the past.

The team agreed that the path forward for Site 17 is a supplemental investigation to address dieldrin and PCBs in soil and groundwater, followed by a FS, PRAP, and ROD.

SAP Worksheet #9-3—Project Scoping Session Participants Sheet

Project Name: Additional Investigations at OU1 Site 17 **Projected Date(s) of Sampling:** Summer 2008 **Project Manager:** Bill Hannah	**Site Name:** 17 **Site Location:** MCAS Cherry Point, North Carolina
Date of Session: October 15, 2007 **Scoping Session Purpose:** To discuss potential sampling locations and development of the work plan and SAP**.**	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Doug Bitterman	Activity Manager, MCAS Cherry Point	CH2M HILL	(757)-671-6209	Doug.Bitterman@CH2M.com	Overseeing UFP-SAP production, Work Plan production, and project support
Bill Hannah	Hydrogeologist	CH2M HILL	(757)-671-6277	Bill.Hannah@CH2M.com	Work Plan production

Comments/Decisions:

Meeting to discuss potential sampling locations and rationale.

Action Items:

Prepare work plan and SAP.

Consensus Decisions:

N/A

SAP Worksheet #9-4—Project Scoping Session Participants Sheet

Project Name: Additional Investigations at OU1 Site 17 **Projected Date(s) of Sampling:** Summer 2008 **Project Manager:** Bill Hannah	**Site Name:** 17 **Site Location:** MCAS Cherry Point, North Carolina
Date of Session: October 24, 2007 **Scoping Session Purpose:** Discuss PCB occurrence at Site 17	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Doug Bitterman	Activity Manager, MCAS Cherry Point	CH2M HILL	(757)-671-6209	Doug.Bitterman@CH2M.com	Overseeing UFP-SAP production, Work Plan production, and project support
Bill Hannah	Hydrogeologist	CH2M HILL	(757)-671-6277	Bill.Hannah@CH2M.com	Work Plan production
Dan Lavoie	Biologist	CH2M HILL	(202) 290-1455	Daniel.Lavoie@CH@M.com	Conducted previous ecological assessments at Site 17

Comments/Decisions:

Conference call conducted to discuss potential sampling locations at Site 17.

Action Items:

N/A

Consensus Decisions:

N/A

SAP Worksheet #9-5—Project Scoping Session Participants Sheet

Project Name: Additional Investigations at OU1 Site 17 **Projected Date(s) of Sampling:** Summer 2008 **Project Manager:** Bill Hannah	**Site Name:** 17 **Site Location:** MCAS Cherry Point, North Carolina
Date of Session: December 4, 2007 **Scoping Session Purpose:** Partnering Meeting	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Janice Nielsen	Remedial Project Manager	NAVFAC Mid-Atlantic	(757) 322-8339	janice.nielsen@navy.mil	Coordinates Environmental Restoration (CERCLA/MRP) activities; Partnering Team member
Jeff Christopher	Installation Restoration Project Manager	MCAS Environmental Affairs Department	Phone: (252) 466-4421	jeffrey.christopher@usmc.mil	Coordinates base environmental activities; Partnering Team member
Gena Townsend	Remedial Project Manager	USEPA Region 4	Phone: (404) 562-8538	townsend.gena@epa.gov	EPA regulator; Partnering Team member
George Lane	Remedial Project Manager	NCDENR	(919) 508-8462	george.lane@ncmail.net	State regulator; Partnering Team member
Doug Bitterman	Activity Manager, MCAS Cherry Point	CH2M HILL	(757)-671-6209	Doug.Bitterman@CH2M.com	Overseeing UFP-SAP production, Work Plan production, and project support; Partnering Team member
Erica DeLattre	Project Manager	Rhea	(724)-443-4111	erica@rhea.us	Partnering Team member
Tim Wenk	Staff Consultant	CH2M HILL	(757)-671-6265	Tim.Wenk@CH2M.com	Drafted meeting minutes
Bill Hannah	Project Manager	CH2M HILL	(757)-671-6277	Bill.Hannah@CH2M.com	Primary author of UFP-SAP

SAP Worksheet #9-5—Project Scoping Session Participants Sheet (continued)

Comments/Decisions:

Bill Hannah/CH2M HILL called in for the discussion since he has been the primary author of the forthcoming work plan.

The work is being conducted to resolve questions that arose about Site 17 during the July 2007 partnering meeting based on findings from the OU1 RI Report (prepared by TetraTech NUS in 2002). Site 17 had dieldrin detections in the shallow soil and in the groundwater above the NC 2L standard. Also, the RI seemed to indicate that there were PCB detections in the soil exceeding the screening criteria despite a removal action in 1995, and there were no groundwater samples analyzed for PCBs.

The CH2M HILL-proposed investigation involved sampling soil and groundwater using direct-push technology (DPT). The proposed sampling strategy consisted of:

- Sampling 1 surface soil sample and 1 groundwater sample each from 4 locations at Site 17 for dieldrin analysis only.
- Sampling 1 surface soil sample and 1 groundwater sample each from 10 locations at Site 17 for PCBs analysis.

Gena stated that the sampling locations should be moved as close as possible to the earlier RI sample locations that had high hits in order to identify if PCB-contaminated areas were missed when the earlier removal action was performed. Doug presented Figures 4-3 and 4-5 from the OU1 RI report and pointed out that the RI report argues that the soils outside of the excavated area were below the 10 ppm action level. The team reviewed the wording in the RI discussing Site 17 soil and agreed the wording in the conclusions for Site 17 is vague and confusing.

After looking at the RI report, Gena said she was comfortable that to the report adequately proved that the excavation was successful in eliminating the risk at the site. She said she wanted to either re-write the RI report paragraph that leads to confusion about the site or to collect additional samples to prove that the RI conclusions with respect to Site 17 PCBs in soil are correct. George indicated he believes collecting new samples is necessary because we do not have any groundwater samples from the site. He said he is not comfortable moving forward without re-sampling the soil and collecting groundwater samples.

Gena said that we need to accept the validity of the data from the RI despite the team's plan to collect additional samples. She believes the purpose of the additional samples is to prove that the removal action was successful or to determine if there are still areas that are above the action level. To prove either point, she wants soil and groundwater samples collected at the same locations as the RI soil sample locations that contained PCB concentrations above 10 mg/kg.

SAP Worksheet #9-5—Project Scoping Session Participants Sheet (continued)

Doug displayed the text in the OU1 RI report stating that the field test kits used in the confirmatory sampling over-estimated the *in situ* concentrations. The team agreed that the data correlation was not very good and that the existing data are not necessarily the best to follow. Therefore, the new sampling will serve the goal of confirming the old data and will address the concerns raised by the team in this discussion. The team agreed that the proposed soil and groundwater sampling locations for dieldrin will be moved to correspond to the locations of the previous soil hits and the PCB sampling locations will be reduced to the 8 locations that correlate to the earlier PCB soil hits above 10 ppm.

Consensus Decisions:

At Site 17, two PCB soil and groundwater sampling locations will be removed from the initially proposed sampling locations at the beginning of the discussion, and that the PCB soil and groundwater samples will be collected at the same locations as the historical "hot spot" locations (over 10 ppm), as per USEPA and NCDENR request.

SAP Worksheet #9-6—Project Scoping Session Participants Sheet

Project Name: Additional Investigations at OU1 Site 17 **Projected Date(s) of Sampling:** Summer 2008 **Project Manager:** Bill Hannah	**Site Name:** 17 **Site Location:** MCAS Cherry Point, North Carolina
Date of Session: February 6, 2008 **Scoping Session Purpose:** Partnering meeting	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Janice Nielsen	Remedial Project Manager	NAVFAC Mid-Atlantic	(757) 322-8339	janice.nielsen@navy.mil	Coordinates Environmental Restoration (CERCLA/MRP) activities
Jeff Christopher	Installation Restoration Project Manager	MCAS Environmental Affairs Department	Phone: (252) 466-4421	jeffrey.christopher@usmc.mil	Coordinates base environmental activities
Gena Townsend	Remedial Project Manager	USEPA Region 4	Phone: (404) 562-8538	townsend.gena@epa.gov	EPA regulator
George Lane	Remedial Project Manager	NCDENR	(919) 508-8462	george.lane@ncmail.net	State regulator
Doug Bitterman	Activity Manager, MCAS Cherry Point	CH2M HILL	(757)-671-6209	Doug.Bitterman@CH2M.com	Overseeing UFP-SAP production, Work Plan production, and project support
Erica DeLattre	Project Manager	Rhea	(724)-443-4111	erica@rhea.us	Partnering Team member
Bill Hannah	Project Manager	CH2M HILL	(757)-671-6277	Bill.Hannah@CH2M.com	Primary author of UFP-SAP; drafted meeting minutes
Sherri Eng	Navy Chemist	NAVFAC Mid-Atlantic	(757)-322-4366		Reviews and approves UFP-SAP; Presented the UFP-SAP process to the Partnering Team
Ed Corl		NAVSEA	(757)-322-4768		Presented the UFP-SAP process to the Partnering Team

SAP Worksheet #9-6—Project Scoping Session Participants Sheet (continued)

Site 17 Discussions and Case Study

The first question posed was to identify who is the lead organization. The answer was the Navy.

The next question was to identify possible contaminants, which are PCBs and dieldrin. It was pointed out that the SAP needs to identify which specific PCBs are the contaminants of concern.

Site 17 is under CERCLA requirements.

Next, the team conducted the problem formulation and development of the conceptual site model. The goal is to close the site and move to a Record of Decision (ROD). There is ambiguity in the historical record as to whether or not the prior removal action resulted in the complete removal of PCB contamination above the action level. The new investigation will determine what is currently there and if land use controls are potentially needed to prevent exposure. PCBs and dieldrin were the COPCs identified from the 2002 RI. The 2002 RI determined that there are no other COPCs at Site 17.

The team then discussed the release mechanisms. The PCBs were a result of a release from a Defense Reutilization and Marketing Office (DRMO) storage pad adjacent to the ditch. No information is available regarding a release mechanism for dieldren, and it may be the result of basewide pesticide application rather than a site-specific release. The media of concern are sediment, soil, and groundwater. The site is industrial land use and the receptors to protect include human – construction worker and future residential and ecological – terrestrial. It is necessary to know the distribution of PCBs and dieldrin. The decision drivers include CERCLA and human health risks.

The CSM was then discussed, which could include a picture, or a wire diagram or written discussion (Worksheet #10). The summary narrative needs to discuss transport mechanisms, receptors, exposure, and site history. Doug had concerns about drawing a picture depicting a dieldrin release as a spill, since it is unknown whether or not dieldrin was actually spilled at Site 17 or simply used for its intended purpose. The CSM drawing could have the effect of cementing the notion that there was a spill. Separate CSMs may be required for PCBs and dieldrin as the distribution is different.

The team then worked on the problem definition. It is not clear if all of the PCB-contaminated soil had been removed in the previous removal action. Are there remaining PCBs left over at the site that could be a potential risk? George also stated that the samples are dated and questioned if the earlier data are even usable. Gena suggested confirming if PCB still occurs at the site above risk-based concentrations.

SAP Worksheet #9-6—Project Scoping Session Participants Sheet (continued)

Sherri questioned the Site 17 boundary and if the team had bounded the contamination. Jan responded that the Site 17 boundary was originally the ditch area, and there is some indication that the contamination is outside the boundary. Doug presented the discussion on PCBs from the 2002 RI report, and how it is unclear and contradictory. Site 17 was chosen for a supplemental investigation as the site still had questions that needed to be answered. Sherri asked if there was any other data research that could resolve this. Jeff responded that we can look through the RCRA documentation to better determine the extent of removal in the 1995 removal action. Doug responded that the excavation areas were not previously surveyed and we have attempted to place on the aerial photograph as accurately as possible. Gena suggested that we are comfortable with the removal area boundaries, but outside the boundaries we would need to rerun the risk evaluation. CERCLA requires a risk-based screening criterion. Gena mentioned that 10 ppm is the action level as the 10-25 ppm range meets the 10-4 to 10-6 risk calculations.

Sherri asked about the nature of the release mechanism for dieldrin. The team did not know the release mechanism, and also discussed that the CERCLA requirements only apply to a spill or disposal activity. She asked if there is a background data set to compare to. The team said that there is no background data set available for pesticides. Doug responded that the background data set was for inorganics, including metals. Gena said we can look at the concentrations and compare to what we routinely see at the base.

Sherri also asked about groundwater contamination, and Doug presented the team with a map presented in the 2002 RI. Gena indicated she would like to see more groundwater data to look for parameters other than PCBs and pesticides. Jan and Doug mentioned that this was not what the team had agreed upon earlier. George also mentioned that he would like us to collect VOC data. Bill presented the VOC plume map that shows that Site 17 is not in the area of the general plume. No decision was made on what parameters should be collected.

Sherri asked what type of samples we are going to need. The team responded with soil samples and Sherri asked at what depths. After some discussion, it was concluded that the samples would be collected from 0 to 1 foot, and the results would be used to confirm previous results and be used for risk screening. Gena was concerned about refining the list of Aroclors in that we may miss analytes. Jan indicated that only the analytes that were previously detected should be included. There was discussion on what the laboratory results indicated from the previous sampling and the likelihood of the additional sampling providing a good set of data with only sampling for analytes previously detected. Gena confirmed that she was satisfied with only measuring the previously detected Aroclors.

Sherri asked what screening criteria the team would use. The team responded that the soil would be screened against the Regional Screening Levels (RSLs). Sherri asked what we are doing with the data set after this screening. The team responded that a point to point comparison would be performed. George also said that we would compare to see if it also supports the older data. Gena said if we compare the data set and have concentrations above the RSLs, “further action” is required. The project team would then reconvene to determine possible future actions, which doesn’t necessarily mean remedial action. If the

new data set doesn't confirm the earlier data, then the older data would not be used. Gena said that 10 ppm is the cleanup level for PCBs, but we would compare to the RSLs (0.74 mg/kg) to determine the next action. Sherri said that the project chemists need to find an analytical method where the detection limit is 3-times below the action limit set by the team.

The team then discussed the management of the data. George only wanted to see validated data. The team then went over the primary objective to verify concentrations, compare to the RSLs, and sample at points with elevated concentrations. If one sample exceeds the screening level, the team will reconvene and decide further action. The team has not typically done a statistical analysis. George said that he did like the statistical analysis approach. Gena said that we would want to redefine the area where we have problems, by comparing to the other data points we have.

SAP Worksheet #9-7—Project Scoping Session Participants Sheet

Project Name: Additional Investigations at OU1 Site 17 **Projected Date(s) of Sampling:** Summer 2008 **Project Manager:** Bill Hannah	**Site Name:** 17 **Site Location:** MCAS Cherry Point, North Carolina
Date of Session: March 19, 2008 **Scoping Session Purpose:** Partnering Meeting	

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Janice Nielsen	Remedial Project Manager	NAVFAC Mid-Atlantic	(757) 322-8339	janice.nielsen@navy.mil	Coordinates Environmental Restoration (CERCLA/MRP) activities
Jeff Christopher	Installation Restoration Project Manager	MCAS Environmental Affairs Department	Phone: (252) 466-4421	jeffrey.christopher@usmc.mil	Coordinates base environmental activities
Gena Townsend	Remedial Project Manager	USEPA Region 4	Phone: (404) 562-8538	townsend.gena@epa.gov	EPA regulator
George Lane	Remedial Project Manager	NCDENR	(919) 508-8462	george.lane@ncmail.net	State regulator
Doug Bitterman	Activity Manager, MCAS Cherry Point	CH2M HILL	(757)-671-6209	Doug.Bitterman@CH2M.com	Overseeing UFP-SAP production, Work Plan production, and project support
Erica DeLattre	Project Manager	Rhea	(724)-443-4111	erica@rhea.us	Partnering Team member
Bill Hannah	Project Manager	CH2M HILL	(757)-671-6277	Bill.Hannah@CH2M.com	Primary author of UFP-SAP; drafted meeting minutes
Tim Wenk	Staff Consultant	CH2M HILL	(757)-671-6265	Tim.Wenk@CH2M.com	Drafted meeting minutes

SAP Worksheet #9-7—Project Scoping Session Participants Sheet (continued)

Site 17 Pesticide Sampling Scoping Session

Bill Hannah led the discussion for completing the UFP-SAP sampling strategy worksheets for the upcoming investigation at Site 17. Worksheets #10 and #11 were presented on the display screen and the team discussed what should go on the form.

During the discussion of how the data will be used, Gena pointed out that this is not a brand new site and there already is a risk assessment that has established a risk at the site. Jeff concurred, but added that risk was generated by grouping several sites together. Doug also added that CH2M HILL re-ran the site-specific risks based on only Site 17 sample data and that a human health risk was identified. The team discussed how to use the new data with respect to risks associated to the site; do we use old and new data, or just new data? Jan said the decision tree/process should specify the path forward – if the data come back with less contamination, then the previous removal action likely removed everything; if the data come back with equal or more contamination, we re-run the risks for the site.

Jeff added that the problem lies in the fact that the 2002 RI indicates that there is an uncertainty with the removal action and whether or not it adequately removed all contaminated soil. Subsequent reviews of the data, however, seem to indicate that higher levels of contamination may exist at the site and the existing/old data are not appropriate for the risk assessment. The apparent discrepancy should be a sufficient basis for re-running the risk assessment at the site with new data.

The dieldrin results from the site are somewhat suspect. Gena said we just need to confirm if there is dieldrin contamination at the site because there are currently some discrepancies in dieldrin data. Therefore, we need to collect soil and groundwater data. Doug said he was concerned about cross-contaminating groundwater by pushing contaminated soil from above into the groundwater during direct-push activities. He said even if we use well-developed pre-packed temporary wells, PCB hits may still be seen since PCBs are hydrophobic and will stay bound to the soil. Gena said she has seen other sites where residual contamination was not an issue unless the levels were very high. Doug said he understood Gena's point but is still concerned about creating liability for the Navy if in reality there is nothing in the groundwater. The team agreed to move forward with the proposed sampling and Doug will do additional research and if problems are identified he will bring this information back to the team.

SAP Worksheet #10—Problem Definition

Site Background

OU1 is an industrial area within the southern portion of MCAS Cherry Point that covers approximately 565 acres (Figures 1 and 2). OU1 comprises more than 70 sites, solid waste management units, and other potential sources of contamination. Site 17 is a 300 feet (ft) long drainage ditch, located in the southeastern portion of OU1, next to the Defense Reutilization and Marketing Office (DRMO) (Figure 2). The ditch is used as part of the Air Station storm drainage system, and drains toward the Runway 5 ditch, which discharges to the Schoolhouse Branch. The adjacent 1-acre area was historically used for material storage that included dichlorodiphenyltrichloroethane (DDT), spent photographic fluid after silver recovery, and PCB-containing transformers.

Release History

PCB-contaminated oil was reportedly drained from transformers to the ditch between 1961 and 1968 (Water & Air Research, 1983). Six transformers, each containing 1,000 gallons of oil, and approximately 100 smaller transformers containing 10 to 500 gallons of oil were reportedly emptied in the drainage ditch (Halliburton NUS, 1993).

Investigation History

A removal action was conducted in 1995 that removed PCB-contaminated soil and sediment to a depth of 1.5 ft and backfilled the excavated areas with clean fill. Confirmation samples collected during the removal action indicated that the PCB-contaminated soil had been excavated.

Historical field investigations conducted after the removal action at Site 17 detected PCBs in shallow and subsurface soil, frequently above screening criteria. Aroclor-1260 was the PCB most often detected, with a maximum soil concentration of 130 mg/kg. No groundwater samples have been analyzed for PCBs at Site 17. Dieldrin was detected in shallow soil and groundwater above the screening criteria. Two shallow soil samples and two groundwater samples contained dieldrin with maximum concentrations of 93 µg/kg and 0.71 µg/L, respectively.

Hydrogeology

Shallow soils beneath Site 17 are generally silty sands to fine-grained sands and groundwater is typically encountered at depths ranging from approximately 6 to 10 feet below ground surface. The first encountered groundwater occurs within the surficial aquifer, which generally flows to the west towards East Prong Slocum Creek. The Yorktown confining unit separates the surficial and Yorktown aquifers, and occurs at an approximate depth of 45 feet below ground surface. Figure 4 presents the hydrogeologic conceptual site model beneath OU1.

Receptors

Potential exposure at Site 17 may occur under current and future land use scenarios. Receptors that may be exposed to Site 17 soil and groundwater include construction workers, future residents, and ecological receptors, such as upper-trophic-level receptors, at Schoolhouse Branch and further downstream at East Prong Slocum Creek.

SAP Worksheet #10—Problem Definition (continued)

General Problems to Address

As part of the 2002 Remedial Investigation at OU1, PCB concentrations were detected above the screening criteria in shallow soil (TetraTech, 2002). As a result, there is uncertainty whether or not the PCB contamination was adequately removed during the previous removal action. In addition, dieldrin concentrations also exceeded screening criteria in shallow soil and groundwater. The source of dieldrin is unknown, and may be related to the DRMO storage area or normal, basewide pesticide applications.

The Partnering Team met in July and December 2007 and February and March 2008, and agreed that additional investigations were needed to characterize the occurrence of dieldrin and PCBs at Site 17, in both shallow soil and groundwater. The Partnering Team consists of representatives of MCAS Cherry Point, NAVFAC Mid-Atlantic, EPA, NCDENR, and support contractors. Site investigations at these sites are implemented under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) procedures.

The objective of this supplemental investigation is to confirm residual contamination of PCBs and dieldrin in shallow soil and groundwater above respective action levels, and to determine if additional investigation or remedial action is necessary at Site 17.

Environmental Questions

Residual soil contamination can continue to be a source of contamination to groundwater, as infiltrating recharge water passes through the contamination. It is not clear if the previous removal action removed all of the PCB-impacted soil above the regulatory action level and if there are any PCB impacts within groundwater. In addition, older historical investigations detected dieldrin above the regulatory standards, and the occurrence of dieldrin is not well understood.

The results of the investigation will be presented in a report.

SAP Worksheet #11—Project Quality Objectives/Systematic Planning Process Statements

- Who will use the data?
  - The data will be used by the Navy and stakeholder agencies (i.e. EPA and NCDENR) to ensure the sites are adequately characterized. If appropriate, measures are taken to provide adequate protection of human health and the environment.
- What are the Project Action Limits (PAL's)? (specific detailed list should be provided in WS#15)
  - PCBs
    - Soil is compared to the 10,000 µg/kg action level (Toxic Substances Control Act [TSCA]/Comprehensive Environmental Response Compensation and Liability Act [CERCLA])
    - Groundwater is compared to 0.5 µg/L (Maximum Contaminant Levels [MCLs]). No State standards exist for PCBs.
  - Dieldrin
    - Soil is compared to 1.13 µg/kg (North Carolina Soil Screening Level [SSL].
    - Groundwater is compared to 0.0022 µg/L (North Carolina 2L Groundwater Quality Standard [NC 2L]).
    - North Carolina Department of Environment and Natural Resources (NCDENR) regulations (15A NCAC 02L.0202 (b)(1)) state that "Where the standard for a substance is less than the practical quantitation limit, the detection of that substance at or above the practical quantitation limit shall constitute a violation of the standard". Therefore, for dieldrin analysis in groundwater, since the NC2L of 0.0022 µg/L is considerably low and therefore not an achievable Laboratory QL, the practical Laboratory QL will become the Project QL Goal. Any detection above 0.01 µg/L will be considered to be an exceedence of the NC2L groundwater standard.
  - A specific detailed list of PALs is provided in Worksheet 15
- What will the data be used for?
  - The data will be used to characterize the presence of PCBs and dieldrin at Site 17, in the soil and groundwater.

SAP Worksheet #11—Project Quality Objectives/Systematic Planning Process Statements (continued)

- What types of data are needed (matrix, target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)?
  - Groundwater and soil samples will be submitted to an EPA and State certified off-site laboratory for analysis.
  - Target analytes are the 3 previously detected PCB aroclors (Aroclor-1248, Aroclor-1254, and Aroclor-1260), and dieldrin.
  - Soil samples will be collected at each location at the surface (0-1 ft bgs) with a stainless steel sampling spoon. Lithologic samples will be collected to the total depth of the boring.
  - Groundwater samples will be collected at the groundwater table using pre-packed temporary monitoring wells installed using Direct-Push Technology (DPT). The temporary wells will be sampled using a low-flow sampling technique and peristaltic pump.
- How "good" do the data need to be in order to support the environmental decision?
  - The data will be of the quantity and quality necessary to provide technically sound and defensible assessments of potential risks to human and ecological receptors posed by the contaminants identified. For risk assessment and high-level decisions, laboratory methods will meet CERCLA, EPA Region 4, and Navy guidance and the data will be validated by a third-party validator using national functional guidance, methodology, and laboratory Standard Operating Procedures (SOPs) as appropriate.
- How much data should be collected (number of samples for each analytical group, matrix, and concentration)?
  - One surface soil sample and one groundwater sample will each be collected at ten locations for PCBs and six locations for dieldrin (based on the March 2008 Partnering Meeting) (Figure 3).
- Where, when, and how should the data be collected/generated?
  - Samples will be collected at Site 17 during one field mobilization event. The field investigation is planned to occur in Summer or Fall 2008.
  - Data will be collected and generated in accordance with the procedures outlined in this UFP-SAP. Specifically, see the SOPs in Appendixes A through C for more details.

SAP Worksheet #11—Project Quality Objectives/Systematic Planning Process Statements (continued)

- Who will collect and generate the data? How will the data be reported?
  - CH2M HILL field staff will collect the samples.
  - Laboratory analysis will be performed by GPL Laboratories.
  - The data report will include a CLP Level IV- equivalent package. This will include a Supplemental Naval Installation Restoration Information Solution Electronic Data Deliverable (SNEDD) deliverable in Microsoft Excel format and a hardcopy of the raw data.
- How will the data be archived?
  - The data will be archived in according to procedures dictated in the Navy CLEAN program/contract. At the end of the project, archived data will be returned to the Navy.
- List the PQOs in the form of if/then qualitative and quantitative statements.
  - The decision tree to be used for the data evaluation during this investigation is presented in Figure 5.
  - If the sample results are below action levels or screening criteria, it will be concluded that the previous removal action was sufficient to allow the team to proceed to site closure. If the concentrations are equal or higher than action levels or screening criteria, PCBs and/or dieldrin will be considered chemicals of potential concern (COPCs), the risks will need to be re-evaluated for the site, and further investigation and/or a remedial action may need to be conducted.

SAP Worksheet #12-1— Measurement Performance Criteria Table

Matrix: Surface Soil

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Equipment Rinsate Blank, Ambient Field Blank	Pesticides	One per day of sampling per type of equipment, one per week	contamination	all target compounds <QL	S & A
Field Duplicate		One per 10 field samples	Precision	should meet RPD criteria of 35% for soil/sediment	S & A
Method Blank		one every batch of 20 samples or less	contamination/bias	all target compounds <QL; surrogates must be within RT windows; surrogate recoveries must be within: Decachlorobiphenyl: 55-130% TCMX: 70-125%	A
Sulfur Blank		one every batch of 20 samples or less	contamination/bias	all target compounds <QL; surrogates must be within RT windows; surrogate recoveries must be within: Decachlorobiphenyl: 55-130% TCMX: 70-125%	A
Instrument Blank		Every 12 hours	contamination/bias	all target compounds <0.5xQL; surrogates must be within RT windows	A
Matrix spike/Matrix spike duplicate, LCS		one every batch of 20 samples or less	Precision/accuracy	Must meet relative RT criteria; Must meet spike recovery criteria between 65-125%. Samples will be spiked with Dieldrin at a concentration of 3.4 ug/kg	A

LCS = Laboratory Control Sample
QL = Quantitation Limit
RPD = Relative Percent Difference
RT = Retention Time

SAP Worksheet #12-2— Measurement Performance Criteria Table

Matrix: Surface Soil

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Equipment Rinsate Blank, Ambient Field Blank	PCBs	One per day of sampling per type of equipment, one per week	contamination	all target compounds <QL	S & A
Field Duplicate		One per 10 field samples	Precision	should meet RPD criteria of 35% for soil/sediment	S & A
Method Blank, Sulfur Blank		one every batch of 20 samples or less	contamination/bias	all target compounds <QL; surrogates must be within RT windows; surrogate recoveries must be within: Decachlorobiphenyl: 60-125%	A
Initial Calibration		before each analytical run	precision/ accuracy	Initial calibration should be done with 5 point calibrations for each PCB of interest.	A
Instrument Blank		Every 12 hours	contamination/bias	all target compounds <0.5xQL; surrogates must be within RT windows	A
Matrix spike/Matrix spike duplicate, LCS		one every batch of 20 samples or less	Precision/accuracy	Must meet relative RT criteria; Must meet spike recovery criteria of 60-130%. Samples will be spiked with Aroclor 1248/1260 mixture at concentrations of 33.4 ug/Kg .	A

LCS = Laboratory Control Sample
QL = Quantitation Limit
RPD = Relative Percent Difference
RT = Retention Time

SAP Worksheet #12-3—Measurement Performance Criteria Table

Matrix: Groundwater

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Equipment Rinsate Blank, Ambient Field Blank	Pesticides	One per day of sampling per type of equipment, one per week	contamination	all target compounds <QL	S & A
Field Duplicate		One per 10 field samples	Precision	should meet RPD criteria of 35% for soil/sediment	S & A
Method Blank		one every batch of 20 samples or less	contamination/bias	all target compounds <QL; surrogates must be within RT windows; surrogate recoveries must be within: Decachlorobiphenyl: 30-135% TCMX: 25-140%	A
Sulfur Blank		one every batch of 20 samples or less	contamination/bias	all target compounds <QL; surrogates must be within RT windows; surrogate recoveries must be within: Decachlorobiphenyl: 30-135% TCMX: 25-140%	A
Instrument Blank		Every 12 hours	contamination/bias	all target compounds <0.5xQL; surrogates must be within RT windows	A
Matrix spike/Matrix spike duplicate, LCS		one every batch of 20 samples or less	Precision/accuracy	must meet relative RT criteria; should meet acceptance criteria and spike recovery criteria of 60-130%. Samples will be spiked with Dieldrin at a concentration of 0.1 ug/L.	A

LCS = Laboratory Control Sample
QL = Quantitation Limit
RPD = Relative Percent Difference
RT = Retention Time

SAP Worksheet #12-4—Measurement Performance Criteria Table

Matrix: Groundwater

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Equipment Rinsate Blank, Ambient Field Blank	PCBs	One per day of sampling per type of equipment, one per week	contamination	all target compounds <QL	S & A
Field Duplicate		One per 10 field samples	Precision	should meet RPD criteria of 35% for soil/sediment	S & A
Method Blank, Sulfur Blank		one every batch of 20 samples or less	contamination/bias	all target compounds <QL; surrogates must be within RT windows; surrogate recoveries must be within: Decachlorobiphenyl: 40-135%	A
Initial Calibration		before each analytical run	Precision/ accuracy	Initial calibration should be done with 5 point calibrations for each PCB of interest.	A
Instrument Blank		Every 12 hours	contamination/bias	all target compounds <0.5 x QL; surrogates must be within RT windows	A
Matrix spike/Matrix spike duplicate, LCS		one every batch of 20 samples or less	Precision/accuracy	Must meet relative RT criteria; Must meet spike recovery criteria of 30-145%. Samples will be spiked with Aroclors 1248/1260 mixture at concentrations of 1 ug/L.	A

LCS = Laboratory Control Sample
QL = Quantitation Limit
RPD = Relative Percent Difference
RT = Retention Time

SAP Worksheet #12-5—Measurement Performance Criteria Table

Matrix: Aqueous

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	TCLP-VOCs	One per batch	Bias / Contamination	No target analytes > Quantitation Limit Surrogates must be within: 1,2-Dichloroethane-d4: 70-120% 4-bromofluorobenzene: 75-120% Dibromofluoromethane: 85-115% Toluene-d8: 85-120%	A
Surrogate Standards		In each sample and QC analysis	Accuracy / Bias	Surrogates must be within: 1,2-Dichloroethane-d4: 70-120% 4-bromofluorobenzene: 75-120% Dibromofluoromethane: 85-115% Toluene-d8: 85-120%	A
Internal Standards		In each sample and QC analysis	Accuracy / Bias / Precision	Area counts –50% to +100% of initial calibration IS or continuing calibration IS area counts; Retention times +/- 30 secs of CC	A
Lab control sample/lab control sample duplicate		One per batch of 20 or fewer samples (include LCSD if no MSD in batch)	Accuracy / Bias / Precision	Benzene: 80-120% Carbon tetrachloride: 65-140% Chlorobenzene: 80-120% Chloroform: 65-135% 1,2-Dichloroethane: 70-130% 1,1-Dichloroethene: 70-130% 2-Butanone: 30-150% Tetrachloroethene: 45-150% Trichloroethene: 70-125% Vinyl Chloride:50-145%	A
Matrix spike/ matrix spike duplicate		One set per 20 field samples	Accuracy / Bias / Precision	Same acceptance criteria as LCS/LCSD	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-6—Measurement Performance Criteria Table

Matrix: Aqueous

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	TCLP-SVOCs	One per batch	Bias / Contamination	No target analytes > Quantitation Limit; Surrogates within: 2-Fluorobiphenyl: 46-108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Nitrobenzene-d5: 38-122%	A
Surrogate Standards		In each sample and QC analysis	Accuracy / Bias	Surrogates within: 2-Fluorobiphenyl: 46-108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Nitrobenzene-d5: 38-122%	A
Internal Standards		In each sample and QC analysis	Accuracy / Bias / Precision	Area counts –50% to +100% of initial calibration IS or continuing calibration IS area counts; Retention times +/- 30 secs of CC	A
Lab control sample/ lab control sample duplicate		One per batch of 20 or fewer samples (include LCSD if no MSD in batch)	Accuracy / Bias / Precision	2-methylphenol: 17-153% 3&4-methylphenol: 21-143% 1,4-Dichlorobenzene: 24-144% 2,4-Dinitrotoluene: 33-153% Hexachlorobenzene: 24-110%Hexachlorobutadiene 25-137% Hexachloroethane: 23-147% Nitrobenzene: 23-147% Pentachlorophenol: 19-110% Pyridine: 23-121% 2,4,5-Trichlorphenol: 28-144% 2,4,6-Trichlorophenol: 31-147%	A
Matrix spike/ matrix spike duplicate		One set per 20 field samples	Accuracy / Bias / Precision	See acceptance criteria for LCS/LCSD.	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-7—Measurement Performance Criteria Table

Matrix: Aqueous

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	TCLP-Pesticides	One per batch	Bias / Contamination	No target analytes > Quantitation Limit; surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%	A
Surrogate Standards		In each sample and QC analysis	Accuracy / Bias	surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%	A
Lab control sample/lab control sample duplicate		One per batch of 20 or fewer samples (include LCSD if no MSD in batch)	Accuracy / Bias / Precision	Endrin: 43-134% Heptachlor: 45-128% Heptachlor epoxide: 53-134% Gamma-BHC: 73-125% Methoxychlor: 73-142%	A
Matrix spike/ matrix spike duplicate		One set per 20 field samples	Accuracy / Bias / Precision	Use acceptance criteria from LCS/LCSD	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-8—Measurement Performance Criteria Table

Matrix: Aqueous

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	TCLP-Herbicides	One per batch	Bias / Contamination	No target analytes > Quantitation Limit; surrogate values within lab statistical QC limits: DCAA: 61-136%	A
Surrogate Standards		In each sample and QC analysis	Accuracy / Bias	DCAA: 61-136%	A
Lab control sample/lab control sample duplicate		One per batch of 20 or fewer samples (include LCSD if no MSD in batch)	Accuracy / Bias / Precision	2,4-D: 61-136% 2,4,5-TP: 61-136%	A
Matrix spike/ matrix spike duplicate		One set per 20 field samples	Accuracy / Bias / Precision	Use acceptance criteria from LCS/LCSD	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-9—Measurement Performance Criteria Table

Matrix: Aqueous

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	TCLP-Metals	One per batch	Bias / Contamination	No target analytes > ½ Quantitation Limit	A
Lab control sample/lab control sample duplicate		One per batch of 20 or fewer samples (include LCSD if no MSD in batch)	Accuracy / Bias / Precision	%Recovery 80% - 120%	A
Matrix spike		One set per 20 field samples	Accuracy / Bias	%Recovery 80% - 120%	A
Post-Digestion Spike		For elements outside of QC limits in matrix spike	Accuracy/Bias	%Recovery 75% - 125%	A
ICP Serial Dilution		Per analytical run for ICP	Accuracy/Bias	%Difference <10%	A
Duplicate		One set per 20 field samples	Precision	Relative Percent Difference <=20%	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-10—Measurement Performance Criteria Table

Matrix: Aqueous

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Lab control sample (pH 7.0 buffer)	Corrosivity	Every 10 samples	Accuracy / Bias	+/- 0.10 pH units	A
Duplicate		One set per 20 field samples	Precision	Relative Percent Difference <=15%	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-11—Measurement Performance Criteria Table

Matrix: Aqueous

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Lab control sample	Ignitability	One per batch of 20 or fewer samples	Accuracy / Bias	%Recovery 80% - 120%	A
Duplicate		One set per 20 field samples, for every sample that flashes, or extinguishes flame <140 degrees	Precision	Relative Percent Difference <=20%	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-12—Measurement Performance Criteria Table

Matrix: Solid

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	TCLP-VOCs	One per batch	Bias / Contamination	No target analytes > Quantitation Limit; surrogates within: 4-bromofluorobenzene: 85-120% Toluene-d8: 85-115%	A
Surrogate Standards		In each sample and QC analysis	Accuracy / Bias	surrogates within: 4-bromofluorobenzene: 85-120% Toluene-d8: 85-115%	A
Internal Standards		In each sample and QC analysis	Accuracy / Bias / Precision	Area counts –50% to +100% of initial calibration IS or continuing calibration IS area counts; Retention times +/- 30 secs of CC	A
Lab control sample/lab control sample duplicate		One per batch of 20 or fewer samples (include LCSD if no MSD in batch)	Accuracy / Bias / Precision	Benzene: 75-125% Carbon tetrachloride: 65-140% Chlorobenzene: 65-135% Chloroform: 70-125% 1,2-Dichloroethane: 70-135% 1,1-Dichloroethene: 65-135% 2-Butanone: 30-160% Tetrachloroethene: 65-140% Trichloroethene: 75-125% Vinyl Chloride:60-125%	A
Matrix spike/matrix spike duplicate		One set per 20 field samples	Accuracy / Bias / Precision	Acceptance criteria from LCS/LCSD	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-13—Measurement Performance Criteria Table

Matrix: Solid

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	TCLP-SVOCs	One per batch	Bias / Contamination	No target analytes > Quantitation Limit; Surrogates within: 2-Fluorobiphenyl: 46-108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Phenol-d5/d6: 34-118% Nitrobenzene-d5: 38-122%	A
Surrogate Standards		In each sample and QC analysis	Accuracy / Bias	Surrogates within: 2-Fluorobiphenyl: 46-108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Phenol-d5/d6: 34-118% Nitrobenzene-d5: 38-122%	A
Internal Standards		In each sample and QC analysis	Accuracy / Bias / Precision	Area counts –50% to +100% of initial calibration IS or continuing calibration IS area counts; Retention times +/- 30 secs of CC	A
Lab control sample/lab control sample duplicate		One per batch of 20 or fewer samples (include LCSD if no MSD in batch)	Accuracy / Bias / Precision	2-methylphenol: 17-153% 3&4-methylphenol: 21-143% 1,4-Dichlorobenzene: 24-144% 2,4-Dinitrotoluene: 33-153% Hexachlorobenzene: 24-110% Hexachlorobutadiene: 25-137% Hexachloroethane: 23-147% Nitrobenzene: 23-147% Pentachlorophenol: 19-110% Pyridine: 23-121% 2,4,5-Trichlorphenol: 28-144% 2,4,6-Trichlorophenol: 31-147%	A
Matrix spike/matrix spike duplicate		One set per 20 field samples	Accuracy / Bias / Precision	Use acceptance criteria from LCS/LCSD	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-14
Measurement Performance Criteria Table

Matrix: Solid

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	TCLP-Pesticides	One per batch	Bias / Contamination	No target analytes > Quantitation Limit; surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%	A
Surrogate Standards		In each sample and QC analysis	Accuracy / Bias	surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%	A
Lab control sample/lab control sample duplicate		One per batch of 20 or fewer samples (include LCSD if no MSD in batch)	Accuracy / Bias / Precision	Endrin: 43-134% Heptachlor: 45-128% Heptachlor epoxide: 53-134% Gamma-BHC: 73-125% Methoxychlor: 73-142%	A
Matrix spike/matrix spike duplicate		One set per 20 field samples	Accuracy / Bias / Precision	Use acceptance criteria from LCS/LCSD	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-15—Measurement Performance Criteria Table

Matrix: Solid

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	TCLP-Herbicides	One per batch	Bias / Contamination	No target analytes > Quantitation Limit; Surrogates within: DCAA: 61-136%	A
Surrogate Standards		In each sample and QC analysis	Accuracy / Bias	Surrogates within: DCAA: 61-136%	A
Lab control sample/lab control sample duplicate		One per batch of 20 or fewer samples (include LCSD if no MSD in batch)	Accuracy / Bias / Precision	2,4-D: 61-136% 2,4,5-TP: 61-136%	A
Matrix spike/matrix spike duplicate		One set per 20 field samples	Accuracy / Bias / Precision	Use acceptance criteria from LCS/LCSD.	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-16—Measurement Performance Criteria Table

Matrix: Solid

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	TCLP-Metals	One per batch	Bias / Contamination	No target analytes > ½ Quantitation Limit	A
Lab control sample/lab control sample duplicate		One per batch of 20 or fewer samples (include LCSD if no MSD in batch)	Accuracy / Bias / Precision	%Recovery 80% - 120%	A
Matrix spike		One set per 20 field samples	Accuracy / Bias	%Recovery 80% - 120%	A
Post-Digestion Spike		For elements outside of QC limits in matrix spike	Accuracy/Bias	%Recovery 75% - 125%	A
ICP Serial Dilution		Per analytical run for ICP	Accuracy/Bias	%Difference <10%	A
Duplicate		One set per 20 field samples	Precision	Relative Percent Difference <=20%	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-17—Measurement Performance Criteria Table

Matrix: Solid

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Lab control sample (pH 7.0 buffer)	Corrosivity	Every 10 samples	Accuracy / Bias	+/- 0.10 pH units	A
Duplicate		One set per 20 field samples	Precision	Relative Percent Difference <=15%	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #12-18—Measurement Performance Criteria Table

Matrix: Solid

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Lab control sample	Ignitability	One per batch of 20 or fewer samples	Accuracy / Bias	%Recovery 80% - 120%	A
Duplicate		One set per 20 field samples, for every sample that flashes, or extinguishes flame <140 degrees	Precision	Relative Percent Difference <=20%	A
Cooler Temperature Indicator		One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees C.	S

SAP Worksheet #13—Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
MCAS Cherry Point Remedial Investigation Data	TetraTech NUS, Inc., *Final Remedial Investigation Operable Unit 1, MCAS Cherry Point, North Carolina*, November 2002	TetraTech NUS, Inc. Soil and groundwater samples collected prior to 2002.	Data used to determine the proposed sample locations for the Work Plan and for comparison against analytical data to be collected.	None known.

SAP Worksheet #14—Summary of Project Tasks

Major tasks associated with the Site 17 sampling effort

The technical approach for the proposed field activities at OU1 is detailed below. The *Final Master Field Sampling Plan, MCAS Cherry Point, North Carolina* (CH2M HILL, 2004) addresses the protocols and standard operating procedures (SOPs) to be used for all investigations at Cherry Point. A site-specific Health and Safety Plan (HSP) to address site-specific details relevant to the Master Field Sampling Plan (FSP) will be completed prior to commencement of the field event.

Mobilization Activities

Prior to mobilization, NAVFAC, MCAS Cherry Point, NCDENR, and EPA will be notified to allow for appropriate oversight and coordination.

As part of the field mobilization, CH2M HILL will procure the following subcontractors to support investigation activities:

- Utility clearance
- Drillers able to provide direct-push soil and groundwater sampling capabilities
- Analytical laboratory
- Data validation
- Investigation-derived waste (IDW) handler with hazardous waste disposal capabilities

Mobilization for the field effort includes procurement of necessary field equipment and initial transport to the site. Equipment and supplies will be brought to the site when the CH2M HILL field team mobilizes for field activities.

Prior to beginning any phase of work, CH2M HILL and its subcontractors will have field meetings to discuss the work items and worker responsibilities, and to familiarize workers with the HSP. Prior to any intrusive activities, the Project Manager (PM) and the Field Team Lead (FTL) will coordinate with Mr. Jeff Christopher, MCAS Cherry Point Environmental Affairs Department (EAD), as well as with critical contacts at Site 17, if applicable. The utilities in the area will be marked out prior to mobilization of the drilling staff. No intrusive activities will be initiated until the utility clearance has been completed.

Direct-Push Technology Sampling

At Site 17, 16 locations will be sampled using DPT, as shown in Figure 3. Details of the sampling are discussed below.

Soil

Soil samples will be collected at each location (10 locations for PCBs and 6 locations for dieldrin) at the surface (0–1 foot bgs) with a stainless steel sampling spoon or trowel. Soil samples will be collected in a jar and submitted for analysis of PCBs or pesticides using methods SW-846 8082 and SW-846 8081A, respectively.

During DPT soil sampling, soil cores will be collected continuously to the final boring depth to characterize the lithology. A CH2M HILL geologist will observe and record soil descriptions that include grain size, color, moisture content, consistency, soil structure,

mineralogy, and other relevant information concerning visible evidence of contamination. All soils collected for analytical or lithologic descriptions will be screened with a photoionization detector (PID) and the levels will be recorded in the field notebook.

Groundwater

Sixteen groundwater samples will be collected (10 samples for PCBs and 6 samples for dieldrin) using temporary monitoring wells with pre-packed sand filter packs that are installed using DPT technology. One groundwater sample will be collected from each temporary well, which will be installed at or across the groundwater table. Each temporary monitoring well will have a 5 foot screen interval. The groundwater will be sampled directly through the screen using a peristaltic pump and low-flow purging techniques (USEPA, 1996) to minimize turbidity, and the approximate sampling depth will be recorded in the log book.

Samples will be contained in laboratory-prepared, pre-preserved sample bottles. Samples will be collected in amber-colored glass bottles, and submitted for analysis of PCBs or pesticides using methods SW-846 8082 and SW-846 8081A, respectively.

Quality Assurance/Quality Control

The quality assurance/quality control (QA/QC) sample collection frequency is as follows (also shown in Worksheet 20):

- **Duplicates:** 1 per 10 samples
- **MS/MSD:** 1 per 20 samples
- **Field Blank:** 1 per week
- **Equipment Blank:** 2 per day (1 soil, 1 groundwater)

Sampling Equipment Decontamination

All non-disposable sampling equipment will be decontaminated immediately after each use in accordance with the applicable SOPs. Heavy equipment such as DPT rods and grab groundwater sampling equipment will be power-washed clean with hot water prior to each new grab groundwater location. A decontamination pad will be set up to prevent the runoff of decontamination water and to allow for easy collection of decontamination fluids.

Investigation-Derived Waste (IDW)

IDW is expected to consist of soil from DPT borings, purge water (from groundwater sampling), and decontamination fluids. Aqueous IDW will be stored in drums and transported to the IWTP for disposal on a daily basis. Soil IDW will be containerized in 55-gallon steel drums and will be labeled appropriately. The soil IDW will be chemically characterized and will be properly disposed of by subcontractors within 90 days of generation. Disposable equipment, including personal protective equipment (PPE), poly sheeting, paper towels, sample tubing, and sampling spoons will be containerized in drums. If soil and groundwater results are determined to be non-hazardous, PPE will be disposed of in trash dumpsters at the base.

Soil and groundwater analysis of the IDW is dependent on the disposal facility's requirements. At a minimum, one soil and one groundwater sample analyzed for pesticides is expected. The full suite of Toxicity Characteristic Leaching Procedure (TCLP) analytes is

included, along with ignitability and corrosivity. If it is determined that only Pesticides are required by the disposal facility, the other TCLP suites will be removed from this SAP.

Sample Analysis and Data Validation

CH2M HILL Genevieve Moore will track the samples from collection through analysis and obtain a Level IV data package from GPL Laboratories within 28 calendar days from sample receipt. All analyses will be conducted at a laboratory that has been reviewed and by Navy Facilities Engineering Service Center (NFESC) personnel (see Worksheet 31). A signed certificate of analysis will be provided in the narrative section of each laboratory data package. The laboratory will submit the data in hard copy and an electronic format. CH2M HILL will manage the data according to the Navy CLEAN Data Management Plan (Appendix C).

Analytical results will be validated from an analytical methodology standpoint by DataQual Environmental Services. Data that should be qualified will be flagged appropriately by incorporating the flagging conventions of the National Functional Guidelines but will use SW846 methodology and measurement performance criteria specified in this SAP. Results for QA/QC samples will be reviewed and the data will be qualified further, if necessary according to QA/QC limits and performance criteria established in SW846 methods 8081A and 8082. Finally, the data set as a whole will be examined for consistency, anomalous results, reasonableness, and utility using professional judgment.

DataQual Environmental Services will be provided with the hard copy and electronic version of the laboratory results and will add data validation qualifiers to both versions. The electronic version will be examined for completeness and accuracy and compared to the hardcopy results by Megan Hilton, project chemist, and then loaded into the CH2M HILL master database.

- **Procedures for recording data, including guidelines for recording and correcting data:**
    - See the *EIS Checklist for Validated and Unvalidated EDDs/SNEDDs and Hard Copy Data Checklist* in Appendix C of this UFP-SAP for examples of CH2M HILL's hardcopy data reporting forms and verification checklists.
    - See the document *SNEDD Specs* in Appendix C for information about CH2M HILL's Navy CLEAN EDD format and valid values.
- **Computerized and manual procedures of data generation to final use and storage and QC checks for error detection to ensure data integrity:**
    - The following documents (found in Appendix C) provide guidance on these procedures:
        - *EIS QC Checklist for Unvalidated and Validated EDDs/ SNEDDs and Hard Copy Data Checklist*
        - *EDD Prep for Raw and Detects Tables for Unvalidated or Validated Data*
        - *EDD Prep for Load and Archive Files*

Guidance on data management steps such as data recording, data transformation, data reduction, data transfer and transmittal, data analysis, and data review can be found in the *Data Management Checklist and Navy CLEAN Data Management Plan* (found in Appendix C).

Procedures for data tracking, storage, archiving, retrieval and security for both electronic and hardcopy data:

- See the *Data Management Checklist, EnStat Instructions*, and *Navy CLEAN Data Management Plan* for more information (Appendix C).
- The project EIS, Genevieve Moore of the CH2M HILL Virginia Beach, VA office, is responsible for data tracking and storage.

Stacy Davenport of the CH2M HILL Chantilly, VA office coordinates archiving and retrieval of data.

SAP Worksheet #15-1—Reference Limits and Evaluation Table

Matrix: Surface Soil

Analytical Group: Pesticides

Analyte	CAS Number	Project Action Limit[1] (ug/kg)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/kg)	Laboratory-specific	
					MDLs (ug/kg)	QLs (ug/kg)
Dieldrin	60-57-1	1.13	North Carolina SSLs	0.57	0.11	0.57

Shading represents Project Action Limits which are below Project Quantitation Limits.

[1] Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Soil results will be compared to North Carolina Soil Screening Levels.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

"**North Carolina SSLs**" are the Soil Screening Levels for the State of North Carolina. These values were calculated in order to protect groundwater for use of drinking.

SAP Worksheet #15-2— Reference Limits and Evaluation Table

Matrix: Surface Soil

Analytical Group: Select PCBs

Analyte	CAS Number	Project Action Limit[1] (ug/kg)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/kg)	Laboratory-specific	
					MDLs (ug/kg)	QLs (ug/kg)
Aroclor-1248	12672-29-6	10000	TSCA/CERCLA	1000	3.8	17
Aroclor-1254	11097-69-1	10000	TSCA/CERCLA	1000	2.0	17
Aroclor-1260	11096-82-5	10000	TSCA/CERCLA	1000	5.3	17

Shading represents Project Action Limits which are below Project Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Soil results will be compared to Toxic Substances Control Act [TSCA]/Comprehensive Environmental Response Compensation and Liability Act [CERCLA].

[3] Project Quantitation Limit Goal was determined by dividing the Project Action Limit by a factor of 10.

"**TSCA/CERCLA**" are soil values given by the Toxic Substances Control Act [TSCA]/Comprehensive Environmental Response Compensation and Liability Act [CERCLA].

SAP Worksheet #15-3— Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: Pesticides

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					MDLs (ug/L)	QLs (ug/L)
Dieldrin	60-57-1	0.0022	North Carolina 2Ls	0.01	0.003	0.01

Shading represents Project Action Limits which are below Project Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Groundwater results will be compared to North Carolina 2 L Groundwater Quality Standards.

[3] North Carolina Department of Environment and Natural Resources (NCDENR) regulations (15A NCAC 02L.0202 (b)(1)) state that “Where the standard for a substance is less than the practical quantitation limit, the detection of that substance at or above the practical quantitation limit shall constitute a violation of the standard”. Therefore, for dieldrin analysis in groundwater, since the NC2L of 0.0022 µg/L is considerably low and therefore not an achievable Laboratory QL, the practical Laboratory QL has become the Project QL Goal. Any detection above 0.01 µg/L will be considered to be an exceedence of the NC2L groundwater standard.

SAP Worksheet #15-4— Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: PCBs

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					MDLs (ug/L)	QLs (ug/L)
Aroclor-1248	12672-29-6	.5	MCLs	0.3	0.14	0.3
Aroclor-1254	11097-69-1	.5	MCLs	0.3	.098	0.3
Aroclor-1260	11096-82-5	.5	MCLs	0.3	0.1	0.3

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Groundwater results will be compared to Federal Maximum Contaminant Levels.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

"**MCLs**" are Federal Maximum Contaminant Levels.

SAP Worksheet #15-5—Reference Limits and Evaluation Table

Matrix: Solid
Analytical Group: TCLP-VOCs (Volatile results from the leaching procedure)

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					QLs (ug/L)	MDLs (ug/L)
1,1-Dichloroethene	75-35-4	700	40 CFR 261.4	100	100	4.2
1,2-Dichloroethane	107-06-2	500	40 CFR 261.4	100	100	7.1
2-Butanone	78-93-3	200000	40 CFR 261.4	100	100	24
Benzene	71-43-2	500	40 CFR 261.4	100	100	. 3.2
Carbon tetrachloride	56-23-5	500	40 CFR 261.4	100	100	7.4
Chlorobenzene	108-90-7	100000	40 CFR 261.4	100	100	4.9
Chloroform	67-66-3	6000	40 CFR 261.4	100	100	5.2
Tetrachloroethene	127-18-4	700	40 CFR 261.4	100	100	3.8
Trichloroethene	79-01-6	500	40 CFR 261.4	100	100	4.4
Vinyl chloride	75-01-4	200	40 CFR 261.4	100	100	9.5

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Solid IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-6—Reference Limits and Evaluation Table

Matrix: Solid
Analytical Group: TCLPS (Semivolatile results from the leaching procedure)

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					QLs (ug/L)	MDLs (ug/L)
2-Methylphenol	95-48-7	200000	40 CFR 261.4	50	50	48
4-Methylphenol	106-44-5	200000	40 CFR 261.4	100	100	44
1,4-Dichlorobenzene	106-46-7	7500	40 CFR 261.4	50	50	6.5
2,4-Dinitrotoluene	121-14-2	130	40 CFR 261.4	50	50	12
Hexachlorobenzene	118-74-1	130	40 CFR 261.4	50	50	9.0
Hexachlorobutadiene	87-68-3	500	40 CFR 261.4	50	50	9.5
Hexachloroethane	67-72-1	3000	40 CFR 261.4	50	50	9.5
Nitrobenzene	98-95-3	2000	40 CFR 261.4	50	50	6.5
Pentachlorophenol	87-86-5	100000	40 CFR 261.4	100	100	22
Pyridine	110-86-1	5000	40 CFR 261.4	50	50	8.5
2,4,5-Trichlorophenol	95-95-4	400000	40 CFR 261.4	50	50	17
2,4,6-Trichlorophenol	88-06-2	2000	40 CFR 261.4	50	50	15

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Solid IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-7—Reference Limits and Evaluation Table

Matrix: Solid
Analytical Group: TCLPP (Pesticide and PCB results from the leaching procedure)

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					QLs (ug/L)	MDLs (ug/L)
gamma-BHC (Lindane)	58-89-9	400	40 CFR 261.4	0.25	0.25	0.010
Heptachlor	76-44-8	8	40 CFR 261.4	0.25	0.25	0.010
Heptachlor epoxide	1024-57-3	8	40 CFR 261.4	0.25	0.25	0.015
Endrin	72-20-8	20	40 CFR 261.4	0.25	0.25	0.012
Methoxychlor	72-43-5	10000	40 CFR 261.4	0.25	0.25	0.016
Toxaphene	8001-35-2	500	40 CFR 261.4	5.0	5.0	0.28
Chlordane (technical)	12789-03-6	30	40 CFR 261.4	5.0	5.0	0.26

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Solid IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-8—Reference Limits and Evaluation Table

Matrix: Solid
Analytical Group: TCLPH (Herbicide result from the leaching procedure)

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					QLs (ug/L)	MDLs (ug/L)
2,4,5-TP (Silvex)	93-72-1	1000	40 CFR 261.4	5.0	5.0	0.32
2,4-D	94-75-7	10000	40 CFR 261.4	5.0	5.0	0.55

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Solid IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-9—Reference Limits and Evaluation Table

Matrix: Solid
Analytical Group: TCLPM (Metal results from the leaching procedure)

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					QLs (ug/L)	MDLs (ug/L)
Arsenic	7440-38-2	5000	40 CFR 261.4	200	200	4.2
Barium	7440-39-3	100000	40 CFR 261.4	1000	1000	32
Cadmium	7440-43-9	1000	40 CFR 261.4	60	60	2.1
Chromium	7440-47-3	5000	40 CFR 261.4	50	50	6.3
Lead	7439-92-1	5000	40 CFR 261.4	100	100	12
Selenium	7782-49-2	1000	40 CFR 261.4	200	200	1.7
Silver	7440-22-4	5000	40 CFR 261.4	50	50	4.7
Mercury	7439-97-6	200	40 CFR 261.4	2.0	2.0	0.19

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Solid IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-10—Reference Limits and Evaluation Table

Matrix: Solid

Analytical Group: CORR (Corrosivity)

Analyte	CAS Number	Project Action Limit[1] (s.u.)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (s.u.)	Laboratory-specific	
					QLs	MDLs
pH	PH	2<pH<12.5	40 CFR 261.4	0<pH<14	0<pH<14	N/A

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Solid IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-11—Reference Limits and Evaluation Table

Matrix: Solid
Analytical Group: IGN (Ignitability)

Analyte	CAS Number	Project Action Limit[1] (degrees F)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (degrees F)	Laboratory-specific	
					QLs (F)	MDLs (F)
Ignitablitity	FLASHPOINT	140	40 CFR 261.4	140	N/A	N/A

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Solid IDW samples will be compared to 40 CFR 261.4.

3 Project Quantitation Limit Goal was determined based on the Project Action Limit.

SAP Worksheet #15-12—Reference Limits and Evaluation Table

Matrix: Liquid
Analytical Group: TCLPV (Volatile results from the leaching procedure)

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					QLs (ug/L)	MDLs (ug/L)
1,1-Dichloroethene	75-35-4	700	40 CFR 261.4	100	100	4.2
1,2-Dichloroethane	107-06-2	500	40 CFR 261.4	100	100	7.1
2-Butanone	78-93-3	200000	40 CFR 261.4	100	100	24
Benzene	71-43-2	500	40 CFR 261.4	100	100	3.2
Carbon tetrachloride	56-23-5	500	40 CFR 261.4	100	100	7.4
Chlorobenzene	108-90-7	100000	40 CFR 261.4	100	100	4.9
Chloroform	67-66-3	6000	40 CFR 261.4	100	100	5.2
Tetrachloroethene	127-18-4	700	40 CFR 261.4	100	100	3.8
Trichloroethene	79-01-6	500	40 CFR 261.4	100	100	4.4
Vinyl chloride	75-01-4	200	40 CFR 261.4	100	100	9.5

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Aqueous IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-13—Reference Limits and Evaluation Table

Matrix: Liquid
Analytical Group: TCLPS (Semivolatile results from the leaching procedure)

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					QLs (ug/L)	MDLs (ug/L)
2-Methylphenol	95-48-7	200000	40 CFR 261.4	50	50	48
4-Methylphenol	106-44-5	200000	40 CFR 261.4	100	100	44
1,4-Dichlorobenzene	106-46-7	7500	40 CFR 261.4	50	50	6.5
2,4-Dinitrotoluene	121-14-2	130	40 CFR 261.4	50	50	12
Hexachlorobenzene	118-74-1	130	40 CFR 261.4	50	50	9
Hexachlorobutadiene	87-68-3	500	40 CFR 261.4	50	50	9.5
Hexachloroethane	67-72-1	3000	40 CFR 261.4	50	50	9.5
Nitrobenzene	98-95-3	2000	40 CFR 261.4	50	50	6.5
Pentachlorophenol	87-86-5	100000	40 CFR 261.4	100	100	22
Pyridine	110-86-1	5000	40 CFR 261.4	50	50	8.5
2,4,5-Trichlorophenol	95-95-4	400000	40 CFR 261.4	50	50	17
2,4,6-Trichlorophenol	88-06-2	2000	40 CFR 261.4	50	50	15

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Aqueous IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-14—Reference Limits and Evaluation Table

Matrix: Liquid
Analytical Group: TCLPP (Pesticide and PCB results from the leaching procedure)

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					QLs (ug/L)	MDLs (ug/L)
gamma-BHC (Lindane)	58-89-9	400	40 CFR 261.4	0.25	0.25	0.010
Heptachlor	76-44-8	8	40 CFR 261.4	0.25	0.25	0.010
Heptachlor epoxide	1024-57-3	8	40 CFR 261.4	0.25	0.25	0.015
Endrin	72-20-8	20	40 CFR 261.4	0.25	0.25	0.012
Methoxychlor	72-43-5	10000	40 CFR 261.4	0.25	0.25	0.016
Toxaphene	8001-35-2	500	40 CFR 261.4	5.0	5.0	0.28
Chlordane (technical)	12789-03-6	30	40 CFR 261.4	5.0	5.0	0.26

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Aqueous IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-15—Reference Limits and Evaluation Table

Matrix: Liquid
Analytical Group: TCLPH (Herbicide results from the leaching procedure)

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					QLs (ug/L)	MDLs (ug/L)
2,4,5-TP (Silvex)	93-72-1	1000	40 CFR 261.4	5.0	5.0	0.32
2,4-D	94-75-7	10000	40 CFR 261.4	5.0	5.0	0.55

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Aqueous IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-16—Reference Limits and Evaluation Table

Matrix: Liquid
Analytical Group: TCLPM (Metal results from the leaching procedure)

Analyte	CAS Number	Project Action Limit[1] (ug/L)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (ug/L)	Laboratory-specific	
					QLs (ug/L)	MDLs (ug/L)
Arsenic	7440-38-2	5000	40 CFR 261.4	200	200	42
Barium	7440-39-3	100000	40 CFR 261.4	1000	1000	32
Cadmium	7440-43-9	1000	40 CFR 261.4	60	60	2.1
Chromium	7440-47-3	5000	40 CFR 261.4	50	50	6.3
Lead	7439-92-1	5000	40 CFR 261.4	100	100	12
Selenium	7782-49-2	1000	40 CFR 261.4	200	200	1.7
Silver	7440-22-4	5000	40 CFR 261.4	50	50	4.7
Mercury	7439-97-6	200	40 CFR 261.4	2.0	2.0	0.19

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Aqueous IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-17—Reference Limits and Evaluation Table

Matrix: Liquid

Analytical Group: CORR (Corrosivity)

Analyte	CAS Number	Project Action Limit[1] (s.u.)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (s.u.)	Laboratory-specific	
					QLs	MDLs
pH	PH	2<pH<12.5	40 CFR 261.4	0<pH<14	0<pH<14	N/A

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Aqueous IDW results will be compared to 40 CFR 261.4.

[3] Project Quantitation Limit Goal was determined based on the Laboratory's achievable Quantitation Limit.

SAP Worksheet #15-18—Reference Limits and Evaluation Table

Matrix: Liquid
Analytical Group: IGN (Ignitability)

Analyte	CAS Number	Project Action Limit[1] (degrees F)	Project Action Limit Reference[2]	Project Quantitation Limit Goal[3] (degrees F)	Laboratory-specific	
					QLs (F)[2]	MDLs (F)
Ignitability	FLASHPOINT	140	40 CFR 261.4	140	N/A	N/A

Shading represents Project Action Limits which are below Laboratory Quantitation Limits.

1 Project Action Limits are based upon the most conservative value found for that particular constituite in applicable regulatory criteria.

[2] Aqueous IDW results will be compared to 40 CFR 261.4.

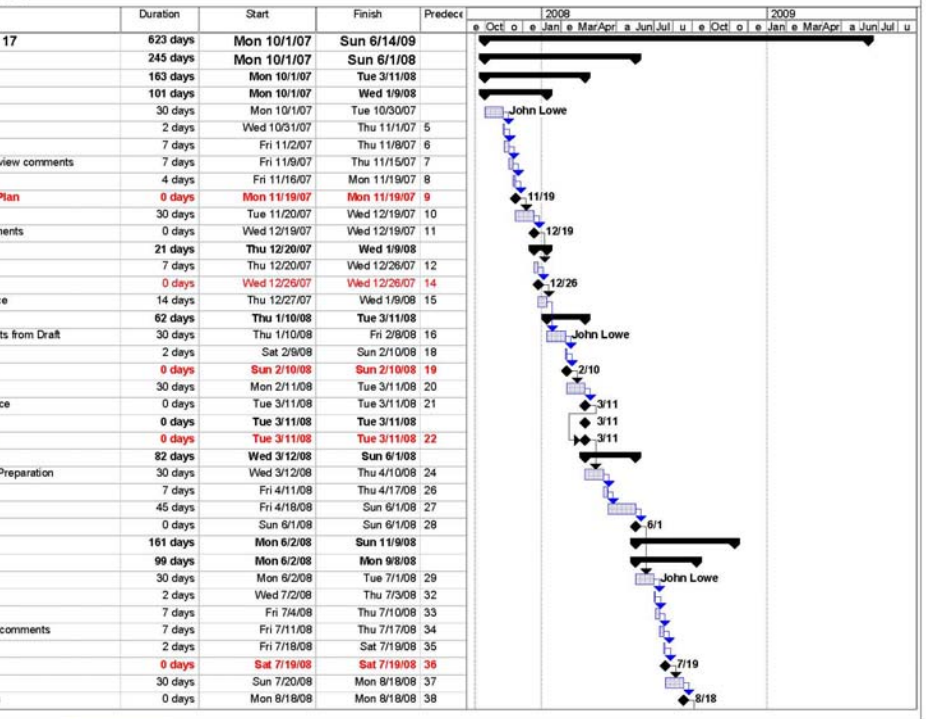
[3] Project Quantitation Limit Goal was determined based on the Project Action Limit.

SAP Worksheet #16—Project Schedule / Timeline Table

The field investigation activities are currently anticipated to occur in the Summer or Fall of 2008. The durations of the field work and investigation reporting are presented below, although the start and end dates have been delayed.

Attachment C
CLEAN III CTO-208 Proposed Schedule
MCAS Cherry Point Sites 15, 16/83, and 17

ID	Task Name	Duration	Start	Finish	Predec
1	CTO-208 - Sites 15, 16/83, and 17	623 days	Mon 10/1/07	Sun 6/14/09	
2	Field Investigation	245 days	Mon 10/1/07	Sun 6/1/08	
3	Work Plan	163 days	Mon 10/1/07	Tue 3/11/08	
4	Draft WP	101 days	Mon 10/1/07	Wed 1/9/08	
5	Prepare WP	30 days	Mon 10/1/07	Tue 10/30/07	
6	editing/formatting	2 days	Wed 10/31/07	Thu 11/1/07	5
7	Senior Review	7 days	Fri 11/2/07	Thu 11/8/07	6
8	Incorporate senior review comments	7 days	Fri 11/9/07	Thu 11/15/07	7
9	Reproduction	4 days	Fri 11/16/07	Mon 11/19/07	8
10	Submit Draft Work Plan	0 days	Mon 11/19/07	Mon 11/19/07	9
11	Team Review	30 days	Tue 11/20/07	Wed 12/19/07	10
12	Receive Team Comments	0 days	Wed 12/19/07	Wed 12/19/07	11
13	RTCs	21 days	Thu 12/20/07	Wed 1/9/08	
14	Prepare RTCs	7 days	Thu 12/20/07	Wed 12/26/07	12
15	Submit RTCs	0 days	Wed 12/26/07	Wed 12/26/07	14
16	Team acceptance	14 days	Thu 12/27/07	Wed 1/9/08	15
17	Draft Final WP	62 days	Thu 1/10/08	Tue 3/11/08	
18	Incorporate Comments from Draft	30 days	Thu 1/10/08	Fri 2/8/08	16
19	Reproduction	2 days	Sat 2/9/08	Sun 2/10/08	18
20	Submit Draft Final	0 days	Sun 2/10/08	Sun 2/10/08	19
21	Regulatory Review	30 days	Mon 2/11/08	Tue 3/11/08	20
22	Regulatory Acceptance	0 days	Tue 3/11/08	Tue 3/11/08	21
23	Final WP	0 days	Tue 3/11/08	Tue 3/11/08	
24	WP becomes Final	0 days	Tue 3/11/08	Tue 3/11/08	22
25	Field Work	82 days	Wed 3/12/08	Sun 6/1/08	
26	Sub Procurement and all Preparation	30 days	Wed 3/12/08	Thu 4/10/08	24
27	Sampling	7 days	Fri 4/11/08	Thu 4/17/08	26
28	Laboratory/DV	45 days	Fri 4/18/08	Sun 6/1/08	27
29	Receive Data	0 days	Sun 6/1/08	Sun 6/1/08	28
30	Investigation Report	161 days	Mon 6/2/08	Sun 11/9/08	
31	Draft Report	99 days	Mon 6/2/08	Mon 9/8/08	
32	Prepare Draft Report	30 days	Mon 6/2/08	Tue 7/1/08	29
33	editing/formatting	2 days	Wed 7/2/08	Thu 7/3/08	32
34	Senior Review	7 days	Fri 7/4/08	Thu 7/10/08	33
35	Incorporate senior review comments	7 days	Fri 7/11/08	Thu 7/17/08	34
36	Reproduction	2 days	Fri 7/18/08	Sat 7/19/08	35
37	Submit Draft Report	0 days	Sat 7/19/08	Sat 7/19/08	36
38	Team Review	30 days	Sun 7/20/08	Mon 8/18/08	37
39	Receive Team Comments	0 days	Mon 8/18/08	Mon 8/18/08	38



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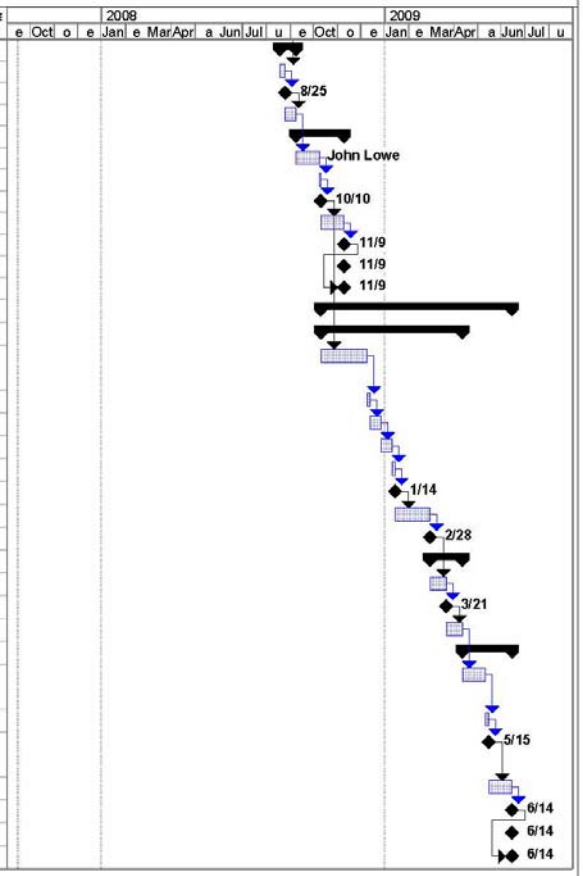
Date Printed: Fri 9/14/07

Task
Split
Progress
Milestone
Summary
Project Summary
External Tasks
External Milestone
Deadline

SAP Worksheet #16—Project Schedule / Timeline Table (continued)

Attachment C
CLEAN III CTO-208 Proposed Schedule
MCAS Cherry Point Sites 15, 16/83, and 17

ID	Task Name	Duration	Start	Finish	Predece
40	**RTCs**	**21 days**	**Tue 8/19/08**	**Mon 9/8/08**	
41	Prepare RTCs	7 days	Tue 8/19/08	Mon 8/25/08	39
42	Submit RTCs	0 days	Mon 8/25/08	Mon 8/25/08	41
43	Team Acceptance	14 days	Tue 8/26/08	Mon 9/8/08	42
44	**Draft Final Report**	**62 days**	**Tue 9/9/08**	**Sun 11/9/08**	
45	Incorporate Comments from Draft	30 days	Tue 9/9/08	Wed 10/8/08	43
46	Reproduction	2 days	Thu 10/9/08	Fri 10/10/08	45
47	Submit Draft Final Report	0 days	Fri 10/10/08	Fri 10/10/08	46
48	Regulatory Review	30 days	Sat 10/11/08	Sun 11/9/08	47
49	Regulatory acceptance	0 days	Sun 11/9/08	Sun 11/9/08	48
50	**Final Report**	**0 days**	**Sun 11/9/08**	**Sun 11/9/08**	
51	Report becomes final	0 days	Sun 11/9/08	Sun 11/9/08	49
52	**Three Separate FSes (same schedules)**	**247 days**	**Sat 10/11/08**	**Sun 6/14/09**	
53	**Three Separate Draft FSes**	**183 days**	**Sat 10/11/08**	**Sat 4/11/09**	
54	Prepare Separate Draft FS Reports for Sites 15, 16/83, and 17	60 days	Sat 10/11/08	Tue 12/9/08	47
55	editing/formatting	4 days	Wed 12/10/08	Sat 12/13/08	54
56	Senior Review	14 days	Sun 12/14/08	Sat 12/27/08	55
57	Incorporate senior review comments	14 days	Sun 12/28/08	Sat 1/10/09	56
58	Reproduction	4 days	Sun 1/11/09	Wed 1/14/09	57
59	Submit Three Draft FSes	0 days	Wed 1/14/09	Wed 1/14/09	58
60	Team Review	45 days	Thu 1/15/09	Sat 2/28/09	59
61	Receive comments	0 days	Sat 2/28/09	Sat 2/28/09	60
62	**RTCs**	**42 days**	**Sun 3/1/09**	**Sat 4/11/09**	
63	Prepare RTCs	21 days	Sun 3/1/09	Sat 3/21/09	61
64	Submit RTCs to Team	0 days	Sat 3/21/09	Sat 3/21/09	63
65	Team acceptance	21 days	Sun 3/22/09	Sat 4/11/09	64
66	**Draft Final FSes**	**64 days**	**Sun 4/12/09**	**Sun 6/14/09**	
67	Incorporate comments from Three Draft FS Reports	30 days	Sun 4/12/09	Mon 5/11/09	65
68	Reproduction	4 days	Tue 5/12/09	Fri 5/15/09	67
69	Submit Three Draft Final FS Reports	0 days	Fri 5/15/09	Fri 5/15/09	68
70	Regulatory Review	30 days	Sat 5/16/09	Sun 6/14/09	69
71	Regulatory acceptance	0 days	Sun 6/14/09	Sun 6/14/09	70
72	**Final FSes**	**0 days**	**Sun 6/14/09**	**Sun 6/14/09**	
73	Three FS Reports become Final	0 days	Sun 6/14/09	Sun 6/14/09	71



Date Revised: 9/14/07 Page 2 of 2
Date Printed: Fri 9/14/07

Task
Split
Progress
Milestone
Summary
Project Summary
External Tasks
External Milestone
Deadline

SAP Worksheet #17—Sampling Design and Rationale

The sampling design and rationale was developed using the Guidance for Performing Site Inspections Under CERCLA (Interim Final, U.S. Environmental Protection Agency, EPA/540-R-92-021, PB92-963375, September 1992) as a reference. The sampling points at each site were located to confirm elevated concentrations detected from previous investigations, to characterize portions of each site not already investigated, and/or to sample for media such as groundwater not previously investigated. The number and locations of the sampling points were discussed and modified with the Partnering Team. The proposed sampling locations are shown in Figure 3.

The quality assurance/quality control (QA/QC) sample collection frequency is as follows:

- **Duplicates:** 1 per 10 samples
- **MS/MSD:** 1 per 20 samples
- **Field Blank:** 1 per week
- **Equipment Blank:** 2 per day (1 soil, 1 groundwater)

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
17DP01 / OU1-17SS01-T-B-MMYY 17DP02 / OU1-17SS02-T-B-MMYY 17DP02 / OU1-17SS02P-T-B-MMYY (duplicate) 17DP03 / OU1-17SS03-T-B-MMYY 17DP04 / OU1-17SS04-T-B-MMYY 17DP05 / OU1-17SS05-T-B-MMYY 17DP06 / OU1-17SS06-T-B-MMYY	Soil	0 – 1-feet	Pesticides, only Dieldrin to be reported	6 field samples 1 field duplicate	ShallowSO
17DP07 / OU1-17SS07-T-B-MMYY 17DP07 / OU1-17SS07P-T-B-MMYY (duplicate) 17DP08 / OU1-17SS08-T-B-MMYY 17DP09 / OU1-17SS09-T-B-MMYY 17DP10 / OU1-17SS10-T-B-MMYY 17DP11 / OU1-17SS11-T-B-MMYY 17DP12 / OU1-17SS12-T-B-MMYY 17DP13 / OU1-17SS13-T-B-MMYY 17DP14 / OU1-17SS14-T-B-MMYY 17DP15 / OU1-17SS15-T-B-MMYY 17DP16 / OU1-17SS16-T-B-MMYY	Soil	0 – 1-feet	PCBs	10 field samples 1 field duplicate	ShallowSO
17DP01 / OU1-17TW01-T-B-MMYY 17DP02 / OU1-17TW02-T-B-MMYY 17DP03 / OU1-17TW03-T-B-MMYY 17DP04 / OU1-17TW04-T-B-MMYY 17DP04 / OU1-17TW04P-T-B-MMYY (duplicate) 17DP05 / OU1-17TW05-T-B-MMYY 17DP06 / OU1-17TW06-T-B-MMYY	Groundwater	Middle of the screen	Pesticides, only Dieldrin to be reported	6 field samples 1 field duplicate	DPGW
17DP07 / OU1-17TW07-T-B-MMYY 17DP08 / OU1-17TW08-T-B-MMYY 17DP09 / OU1-17TW09-T-B-MMYY 17DP10 / OU1-17TW10-T-B-MMYY 17DP11 / OU1-17TW11-T-B-MMYY 17DP12 / OU1-17TW12-T-B-MMYY 17DP13 / OU1-17TW13-T-B-MMYY 17DP14 / OU1-17TW14-T-B-MMYY 17DP14 / OU1-17TW14P-T-B-MMYY (duplicate) 17DP15 / OU1-17TW15-T-B-MMYY 17DP16 / OU1-17TW16-T-B-MMYY	Groundwater	Middle of the Screen	PCBs	10 field samples 1 field duplicate	DPGW
OU1-IDA-MMYY	Aqueous	Composite	Full TCLP, Corrosivity, Ignitability	1 IDW sample	HSE-408, 411
OU1-IDS-MMYY	Solid			1 IDW sample	

[1]Standard operating procedure (SOP) or worksheet listed in Worksheet #21 that describes the sample collection procedures.
All samples will be named in accordance with sample nomenclature scheme “Cherry Point SN” included in Appendix B.
T – Top Depth, B – Bottom Depth, IDA – Aqueous Investigation Derived Waste, IDS – Solid Investigation Derived Waste, MMYY – Month and Year sample collected

SAP Worksheet #19—Analytical SOP Requirements Table

Matrix	Analytical Group	Analytical and Preparation Method/SOP Reference[1]	Sample Volume	Containers (Number, Size, and Type)	Preservation Requirements (Chemical, Temperature, Light Protected)	Maximum Holding Time (Preparation/Analysis)[1]
GW	Pesticides	SW-846 8081A/ Q.6	1 L	1 L amber glass with Teflon-lined cap	cool to 4±2 degrees C	7 days of sample collection to extract/40 days analysis
	PCBs	SW-846 8082/ Q.7	1 L	1 L amber glass with Teflon-lined cap	cool to 4±2 degrees C	7 days of sample collection to extract/40 days analysis
SS	Pesticides	SW-846 8081A/ Q.6	250 GM	8oz glass with Teflon lined cap	cool to 4±2 degrees C	14 days of sample collection to extract/40 days analysis
	PCBs	SW-846 8082/ Q.7	250 GM	8oz glass with Teflon lined cap	cool to 4±2 degrees C	14 days of sample collection to extract/40 days analysis
Aqueous IDW	TCLP Volatiles	SW-846 1311, 8260B/ H.7, M.5	1L	1 L amber glass with Teflon-lined cap	cool to 4±2 degrees C	7 days of sample filtration to analyze
	TCLP Semivolatiles	SW-846 1311, 8270C/ H.7, P.5	1L	1 L amber glass with Teflon-lined cap	cool to 4±2 degrees C	7 days of sample filtration to extract/40 days analysis
	Corrosivity	SW-846 9045C/ J.12	100ml	125ml HDPE	cool to 4±2 degrees C	24 hours to analyze
	Ignitability	Pensky Martens/ N.1	100ml	125ml HDPE	cool to 4±2 degrees C	28 days to analyze
	TCLP Herbicides	SW-846 1311, 8151A/ H.7, Q.10	1L	1 L amber glass with Teflon-lined cap	cool to 4±2 degrees C	7 days of sample filtration to extract/40 days analysis
	TCLP Metals	SW-846 1311, 6010B, 7470A/ H.7, H.10, H.12	1L	1 L HDPE	cool to 4±2 degrees C	6 months
	TCLP Pesticides	SW-846 1311, 8081A/ H.7, Q.6	1L	1 L amber glass with Teflon-lined cap	cool to 4±2 degrees C	7 days of sample filtration to extract/40 days analysis
Solid IDW	TCLP Volatiles	SW-846 1311, 8260B/ H.7, M.5	100g	4oz clear glass wide-mouth with Teflon-lined cap	cool to 4±2 degrees C	7 days of sample extract filtration to analysis
	TCLP Semivolatiles	SW-846 1311, 8270C/ H.7, P.5	100g	8oz clear glass wide-mouth with Teflon-lined cap	cool to 4±2 degrees C	7 days of sample extract filtration to extract/40 days analysis
	Corrosivity	SW-846 9045C/ J.12	20g	4oz clear glass wide-mouth with Teflon-lined cap	cool to 4±2 degrees C	28 days to analyze
	Ignitability	Pensky Martens/ N.1	20g	4oz clear glass wide-mouth with Teflon-lined cap	cool to 4±2 degrees C	28 days to analyze
	TCLP Herbicides	SW-846 1311, 8151A/ H.7, Q.10	100g	8oz clear glass wide-mouth with Teflon-lined cap	cool to 4±2 degrees C	7 days of sample extract filtration to extract/40 days analysis
	TCLP Metals	SW-846 1311, 6010B, 7471A/ H.7, H.10, H.12	100g	8oz clear glass wide-mouth with Teflon-lined cap	cool to 4±2 degrees C	6 months to analyze extract
	TCLP Pesticides	SW-846 1311, 8081A/ H.7, Q.6	100g	8oz clear glass wide-mouth with Teflon-lined cap	cool to 4±2 degrees C	7 days of sample extract filtration to extract/40 days analysis

[1] Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/ extracted. (Not VTSR)

SAP Worksheet #20—Field Quality Control Sample Summary Table

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks	No. of PT Samples	Total No. of Samples to Lab
Surface Soil	PCBs	10	1	1/1	1	1	0	0	14
Groundwater	PCBs	10	1	1/1	1	1	0	0	14
Surface Soil	Pesticides	6	1	1/1	1	1	0	0	9
Groundwater	Pesticides	6	1	1/1	1	1	0	0	9
Aqueous IDW	Full TCLP, Corrosivity, Ignitability	1	0	0	0	0	0	0	1
Solid IDW	Full TCLP, Corrosivity, Ignitability	1	0	0	0	0	0	0	1

SAP Worksheet #21—Project Sampling SOP References Table

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
DPGW	DIRECT-PUSH GROUNDWATER SAMPLE COLLECTION, 5/20/03	CH2M HILL	Geoprobe sampling rods, slotted lead rod, sample containers	N	
DPSoil	Direct-Push Soil Sample Collection, 5/16/03	CH2M HILL	Truck-mounted hydraulic pressure hammer, sampling rods and tubes, acetate sleeves, sample containers	N	
ShallowSO	Shallow Soil Sampling, 5/16/03	CH2M HILL	Hand auger, spatula, measuring tape, pin flags, sample containers	N	
EquipClean	Equipment Cleaning	CH2M HILL	DI Water	N	
FieldMeas	Field Measurements	CH2M HILL	Thermometer, pH meter, SEC meter	N	
MiniRam	Miniram Personal Monitor, 5/16/03	CH2M HILL	Miniram, calibration kit	Y	The MiniRAE 2000 will be used for this project. See the Manufacturer's Instructions, Appendix B, for more details.
HSE-408	Waste Management: Analysis and Characterization, 10/11/07	CH2M HILL	Field logbook, Chain of Custody, sample labels, custody seals	N	
HSE-411	Waste Management: Non-Hazardous Waste, 10/12/07	CH2M HILL	Container labels, waste containers,	N	
HSE-412	Waste Management: PCBs, 10/15/07	CH2M HILL	Container labels	N	

Field SOPs are included in Appendix B.

SAP Worksheet #22—Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Activity[1]	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference[2]	Comments
Horiba U-22 pH probe	Calibration	Daily, before use	pH reads 4.0 +/- 3%	Clean probe with Deionized water and calibrate again. Do not use instrument if not able to calibrate properly	Field Team Lead	HoribaU22	
Horiba U-22 Specific conductance probe	Calibration	Daily, before use	Conductivity reads 4.49 +/- 3%	Clean probe with deionized water and calibrate again. Do not use instrument if not able to calibrate properly.	Field Team Lead	HoribaU22	
Horiba U-22 Turbidity probe	Calibration	Daily, before use	Turbidity reads 0 +/- 3%	Clean probe with deionized water and calibrate again. Do not use instrument if not able to calibrate properly.	Field Team Lead	HoribaU22	
Horiba U-22 Dissolved oxygen and Temperature Probes	Testing	Daily, before use	Consistent with the current atmospheric pressure and ambient temperature	Clean probe with deionized water and calibrate again. Do not use instrument if not able to calibrate properly.	Field Team Lead	HoribaU22	
Horiba U-22	Maintenance- Check mechanical and electronic parts, verify system continuity, check battery, and clean probes. Calibration check	Daily before use, at the end of the day, and when unstable readings occur.	Stable readings after 3 minutes. pH reads 4.0 +/- 3% conductivity reads 4.49 +/- 3% turbidity reads 0 +/- 3%	Clean probe with deionized water and calibrate again. Do not use instrument if not able to calibrate properly.	Field Team Lead	HoribaU22	
MiniRAE 2000	Activities are described in the MiniRAE 2000 Manufacturer's Instructions, provided in Appendix B						

[1] Activities may include: calibration, verification, testing, and maintenance.

[2] Reference from the Project Sampling SOP References table (Worksheet #21).

SAP Worksheet #23—Analytical SOP References Table

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
Q7	SW-846 8082A, October 07, Rev. 8	Definitive	Select PCBs	GC/ECD	GPL Laboratories, LLLP	N
Q6	SW-846 8081, October 07, Rev. 11		Pesticides (Dieldrin)	GC/ECD		
F2	Sample Receipt, Inspection, Preservation, and Storage Condition Requirements, September 07, Rev. 18		Sample Management	N/a		
H7	Toxicity Characterization Leaching Procedure (TCLP) , October 06, Rev. 9		TCPL Organics/Inorganics	N/A		
M5	SW-846 8260B, August 07, Rev. 18		TCLP Volatiles	GC/MS		
P5	SW-846 8270C, August 07, Rev. 15		TCLP Semivolatiles	GC/MS		
Q6	SW-846 8081A, August 07, Rev. 12		TCLP Pesticides	GC/ECD		
Q10	SW-846 8151A, December 05, Rev. 7		TCLP Herbicides	GC/ECD		
H10	SW-846 6010B, November 07, Rev. 18		TCLP Metals	ICP		
H12	SW-846 7470A, September 07, Rev. 22		TCLP Metals	Mercury Analyzer		
N1	SW-846 1010, February 07, Rev. 8	Definitive	Ignitability	Flashpoint Analyzer	GPL Laboratories, LLP	N
J12	SW-846 7.2.2-1a, August 04, Rev. 5		Corrosivity (pH)	pH meter		

Analytical SOPs are included in Appendix A.

SAP Worksheet #24—Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC-ECD PCBs	5 point calibration plus ICV	CCV every 10 samples	%RSD <20% for initial curve Aroclors, %difference from ICAL <25% for CCV for individual Aroclors, not to exceed average of 15% for all Aroclors	correct problem and rerun	Rekha Patel	Q7
GCMS Semivolatiles	Minimum five point calibration for all analytes	Daily, Prior to sample analysis or instrument change, when instrument does not meet method criteria	30% RSD for CCC's and Min RF for SPCCs, 15% for Avg RF, 0.995 corr for linear, 0.99 corr for Quadratic	Recalibrate and or perform necessary instrument maintenance, Check calibration standards, Reanalyze affected samples	Hall Moore	P5
GCMS Volatiles	Minimum five point calibration for all analytes	Daily, Prior to sample analysis or instrument change, when instrument does not meet method criteria	30% RSD for CCC's and Min RF for SPCCs, 15% for Avg RF, 0.995 corr for linear, 0.99 corr for Quadratic	Recalibrate and or perform necessary instrument maintenance, Check calibration standards, Reanalyze affected samples	Nathan Krueger	M5
GC-ECD Pesticides	5 point calibration plus ICV	CCV every 10 samples	%RSD <20% for initial curve, %difference from ICAL <15% for CCV	correct problem and rerun	Rekha Patel	Q6
GC-ECD Herbicides	5 point calibration plus ICV	CCV every 10 samples	%RSD <20% for initial curve, %difference from ICAL <15% for CCV	correct problem and rerun	Rekha Patel	Q10
pH Meter	Calibrate meter at pH 10 and 4, check 7 4 and 10 alternately as appropriate to pH of samples	Before analysis and check every 3 hrs or 10 samples	+/- 0.10 pH units for every check	Recalibrate as necessary	James Anderson	J.12

SAP Worksheet #24—Analytical Instrument Calibration Table (continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
ICP	One point calibration per manufacturers guidelines	At the beginning of each day or if QC is outside criteria	90-110% of true value	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Rita Amin	H.10
UV/Vis Spectrophotometer	Minimum five point calibration	At the beginning of each day or if QC is outside criteria	Correlation coefficient >0.995	Recalibrate and or perform necessary instrument maintenance, Check calibration standards, Reanalyze affected samples	James Anderson	J.43
Hg Analyzer/FIMS	Minimum five point calibration	At the beginning of each day or if QC is outside criteria	Correlation coefficient >0.995	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Rita Amin	H.12
Flashpoint Tester	Flashpoint of p-xylene	At the beginning and end of each set of 20 samples or less	Flash at 27 degrees C, + 2.2 degrees C	Check standard	Namory Keita	N.1

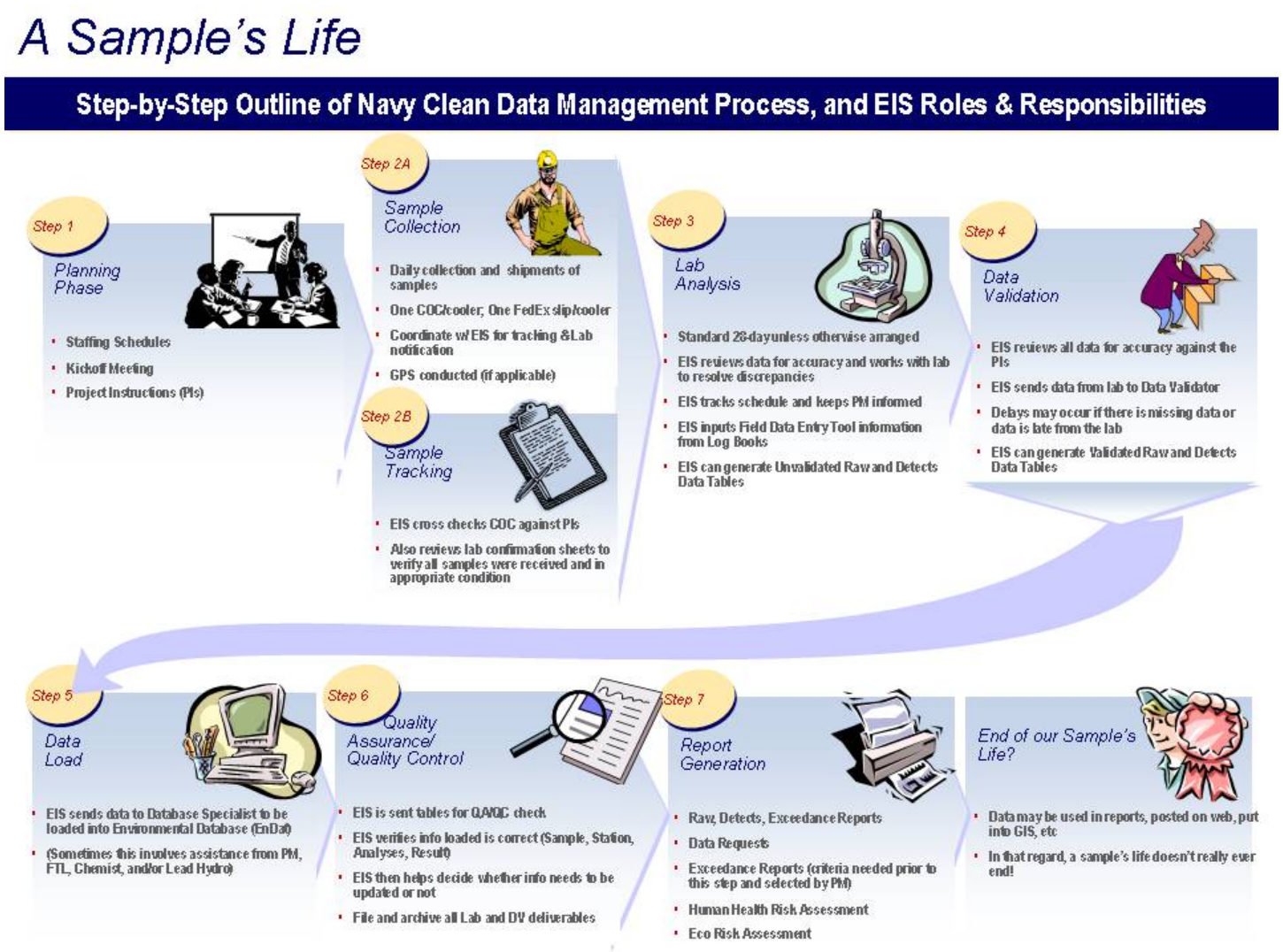
SAP Worksheet #25—Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GCMS	Check gas supply daily, Bake or change trap as necessary, Manual tune if BFB/DFTPP not within criteria, Cut column, change septum as needed	VOA/ SVOA Analysis	Ion source, seal septum, liner	Prior to sample analysis or, when instrument does not meet method criteria	30% RSD CCCs, min RF SPCCs, 15% Avg RSD, 0.995 linear, 0.99 corr. Quadratic init cal; 20% diff CCV for CCCs, min RF SPCCs	Recalibrate and or perform necessary instrument maintenance, Check calibration standards, Reanalyze affected samples	Nathan Krueger/Hall Moore	M5, P5
GC/ECD	change septum and liner, trim analytical column	Calibration	CCV analysis	Daily	Acceptable chromatography and %Difference	repeat	Rekha Patel	Q6, Q7
pH meter	Change buffer solutions or pH probe	Calibration	Calibration Check	Before analysis begins, check every 3 hrs	pH within +/- 0.10 of buffer value	Recalibrate as necessary	James Anderson	J.12
UV/Vis Spectrophotometer	check wavelength, prepare fresh coloring reagents	Calibration	Calibration Check	At the beginning of each day or when QC is outside criteria	Correlation coefficient >0.995	Recalibrate and/or Check calibration standards, prepare fresh color reagents	James Anderson	J.43
Hg Analyzer	Change tubing, change filter, clean windows, check gas flow, Check reagents and standards	Hg Analysis	Change tubing, change filter, clean windows, check gas flow, Check reagents and standards	At the beginning of each day or when QC is outside criteria	Correlation coefficient >0.995	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Rita Amin	H.12
Flashpoint Tester	Change propane tank, calibrate thermometer	Flashpoint	Tank, thermometer	Before use	Flash at 27 degrees C, + 2.2 degrees C	Check standard	Namory Keita	N.1

SAP Worksheet #26-1—Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Project Field Team, FTL/CH2M HILL. Field SOPs are in Appendix B of this SAP.
Sample Packaging (Personnel/Organization): Project Field Team, FTL/CH2M HILL. Field SOPs are in Appendix B of this SAP.
Coordination of Shipment (Personnel/Organization): FTL/CH2M HILL
Type of Shipment/Carrier: FedEx Priority Overnight
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): GPL Laboratories personnel (TBD)
Sample Custody and Storage (Personnel/Organization): GPL Laboratories personnel (TBD)
Sample Preparation (Personnel/Organization): GPL Laboratories personnel (TBD)
Sample Determinative Analysis (Personnel/Organization): GPL Laboratories personnel (TBD)
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 90 days from receipt
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 1 year
Biological Sample Storage (No. of days from sample collection): n/a
SAMPLE DISPOSAL
Personnel/Organization: GPL Laboratories personnel (TBD)
Number of Days from Analysis: After submission, the laboratory will keep samples 90 days and the sample extracts for a minimum of 60 days.

SAP Worksheet #26-2—Sample Handling Flow Diagram
Navy CLEAN Data Management Process



SAP Worksheet #27—Sample Custody Requirements Table

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):

Samples will be collected by field team members under the supervision of the field team leader. As samples are collected, they will be places into containers and labeled. Labels will be taped to the jar to ensure they do not separate. Samples will be cushioned with packaging material and placed into coolers containing enough ice to keep the samples 4 +/- 2 degrees Celsius until they are received by the laboratory.

The chain of custody will be placed into the cooler in a Ziploc bag. Coolers will be taped up and shipped to the laboratories via Fed Ex overnight, with the air bill number indicated on the Chain of Custody (COC) (to relinquish custody). Upon delivery, the laboratory will log in each cooler and report the status of the samples to CH2M HILL. An example COC, custody seals, and sample labels are included in Appendix B.

See Worksheet 21 for SOPs containing sample custody guidance.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):

Laboratory custody procedures can be found in the following SOPs, which are referenced in Worksheet 23 and can be found in Appendix A of this SAP:

GPL Labs: F2

Sample Identification Procedures:

Sample labels will include, at a minimum, client name, site, sample ID, date/time collected, analysis group or method, and sampler's initials. The field logbook will identify the sample ID with the location and time collected and the parameters requested. The laboratory will assign each field sample a laboratory sample ID based on information in the chain of custody. The laboratory will send sample log-in forms to the EIS to check that sample IDs and parameters are correct.

Chain-of-custody Procedures:

Chain of custodies will include, at a minimum, laboratory contact information, client contact information, sample information, and relinquished by/received by information. Sample information will include sample ID. Date/time collected, number and type of containers, preservative information, analysis method, and comments. The chain of custody will link location of the sample from the field logbook to the laboratory receipt of the sample. The laboratory will use the sample information to populate the LIMS database for each sample.

SAP Worksheet #28-1— QC Samples Table

Matrix	Surface Soil
Analytical Group	Pesticides (Dieldrin)
Analytical Method/SOP Reference	SW-846 8081A/ Q6

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	one every batch of 20 samples or less	concentrations of all compounds must be <1/2RL must meet acceptance criteria for surrogates: Decachlorobiphenyl: 55-130% TCMX: 70-125%	reanalysis or reextraction	Veena Telhan/Rehka Patel	contamination/bias	the target analytes must be <0.5xRL must meet acceptance criteria for surrogates: Decachlorobiphenyl: 55-130% TCMX: 70-125%
Field Duplicate	as needed	RPD<35%	Document	Rehka Patel	precision	should meet RPD criteria of 35%
MS/MSD	one set every 20 samples or less	%Recoveries within 65-125%	document matrix interference or reextraction	Veena Telhan/Rehka Patel	precision/accuracy	Must meet relative RT criteria; should meet RPD criteria of 35%; Must meet spike recovery criteria between 65-125%. Samples will be spiked with Dieldrin at a concentration of 3.4 ug/kg
LCS	one set every 20 samples or less	%Recoveries within 65-125%	reanalysis or reextraction	Veena Telhan/Rehka Patel	accuracy	Must meet spike recovery criteria between 65-125%. Samples will be spiked with Dieldrin at a concentration of 3.4 ug/kg
PEM – Breakdown Check	Daily	Breakdown of 4,4'-DDT and Endrin must be less than 15%	Clip column, clean injection port and reanalyze	Rehka Patel	contamination	<15% breakdown of 4,4'DDT and Endrin
Instrument Blank	every 12 hours	concentrations of all compounds must be <1/2RL	reanalysis	Rehka Patel	contamination	target analytes must be <0.5xRL

MS = Matrix Spike
MSD = Matrix Spike Duplicate
LCS = Laboratory Control Sample
PEM = Performance Evaluation Mixture
RL = Reporting Limit
RPD = Relative Percent Difference

SAP Worksheet #28-2— QC Samples Table

Matrix	Surface Soil
Analytical Group	Select PCBs
Analytical Method/SOP Reference	SW-846 8082/Q7

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	one every batch of 20 samples or less	concentrations of all compounds must be <1/2RL must meet acceptance criteria for surrogates: Decachlorobiphenyl: 60-125%	reanalysis or reextraction	Veena Telhan/Rehka Patel	contamination/bias	the target analytes must be <0.5xCRQL; must meet acceptance criteria for surrogates: Decachlorobiphenyl: 60-125%
Field Duplicate	One per 10 field samples	RPD<35%	Document	Rehka Patel	precision	should meet RPD criteria of 35%
Initial Calibration Standards	At beginning of analysis	RSD<20% for five point calibration	recalibrate	Veena Telhan/Rehka Patel	Precision/accuracy	Must perform 5 pt calibration for each PCB of interest.
MS/MSD	one set every 20 samples or less	%Recoveries within 60-130%	document matrix interference or reextraction	Veena Telhan/Rehka Patel	precision/accuracy	Must meet relative RT criteria; should meet RPD criteria of 35%; Must meet spike recovery criteria of 60-130%. Samples will be spiked with Aroclor 1248/1260 mixture at concentrations of 33.4 ug/Kg
LCS	one set every 20 samples or less	%Recoveries within 60-130%	reanalysis or reextraction	Veena Telhan/Rehka Patel	accuracy	Must meet spike recovery criteria of 60-130%. Samples will be spiked with Aroclor 1248/1260 mixture at concentrations of 33.4 ug/Kg

MS = Matrix Spike
MSD = Matrix Spike Duplicate
LCS = Laboratory Control Sample
CRQL = Contract Required Quantitation Limit
RPD = Relative Percent Difference

SAP Worksheet #28-3—QC Samples Table

Matrix	Groundwater
Analytical Group	Pesticides (Dieldrin)
Analytical Method/SOP Reference	SW-846 8081A/Q6

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	one every batch of 20 samples or less	concentrations of all compounds must be <1/2RL must meet acceptance criteria for surrogates: Decachlorobiphenyl: 30-135% TCMX: 25-140%	reanalysis or reextraction	Veena Telhan/Rehka Patel	contamination/bias	the target analytes must be <0.5xCRQL; must meet acceptance criteria for surrogates: Decachlorobiphenyl: 30-135% TCMX: 25-140%
Field Duplicate	as needed	RPD<35%	Document	Rehka Patel	precision	should meet RPD criteria of 35%
MS/MSD	one set every 20 samples or less	%Recoveries within 60-130%	document matrix interference or reextraction	Veena Telhan/Rehka Patel	precision/accuracy	Must meet relative RT criteria; should meet RPD criteria of 35%; should meet acceptance criteria and spike recovery criteria of 60-130%. Samples will be spiked with Dieldrin at a concentration of 0.1 ug/L.
LCS	one set every 20 samples or less	%Recoveries within 60-130%	reanalysis or reextraction	Veena Telhan/Rehka Patel	accuracy	Should meet acceptance criteria and spike recovery criteria of 60-130%. Samples will be spiked with Dieldrin at a concentration of 0.1 ug/L.
Instrument Blank	every 12 hours	concentrations of all compounds must be <1/2RL	reanalysis	Rehka Patel	contamination	target analytes must be <0.5RL
PEM – Breakdown Check	Daily	Breakdown of 4,4'-DDT and Endrin must be less than 15%	Clip column, clean injection port and reanalyze	Rehka Patel	contamination	<15% breakdown of 4,4'DDT and Endrin

MS = Matrix Spike
MSD = Matrix Spike Duplicate
LCS = Laboratory Control Sample
RL = Reporting Limit
RPD = Relative Percent Difference

SAP Worksheet #28-4—QC Samples Table

Matrix	Groundwater
Analytical Group	Select PCBs
Analytical Method/SOP Reference	SW-846 8082/Q7

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	one every batch of 20 samples or less	concentrations of all compounds must be <1/2RL must meet acceptance criteria for surrogate: Decachlorobiphenyl: 40-135%	reanalysis or reextraction	Veena Telhan/Rehka Patel	contamination/bias	the target analytes must be <0.5xRL; must meet acceptance criteria for surrogate: Decachlorobiphenyl: 40-135%
Field Duplicate	as needed	RPD<35%	Document	Rehka Patel	precision	should meet RPD criteria of 35%
Initial Calibration Standards	At beginning of analysis	RSD<20% for five point calibration	recalibrate	Veena Telhan/Rehka Patel	Precision/accuracy	Must perform 5 pt calibration for each PCB of interest.
MS/MSD	one set every 20 samples or less	%Recoveries within 30-145%	document matrix interference or reextraction	Veena Telhan/Rehka Patel	precision/accuracy	Must meet relative RT criteria; should meet RPD criteria of 35%; Must meet spike recovery criteria of 30-145%. Samples will be spiked with Aroclors 1248/1260 mixture at concentrations of 1 ug/L.
LCS	one set every 20 samples or less	%Recoveries within 30-145%	reanalysis or reextraction	Veena Telhan/Rehka Patel	accuracy	Must meet relative RT criteria; Must meet spike recovery criteria of 30-145%. Samples will be spiked with Aroclors 1248/1260 mixture at concentrations of 1 ug/L.

LCS = Laboratory Control Sample
RL = Reporting Limit

MS = Matrix Spike
MSD = Matrix Spike Duplicate
RPD = Relative Percent Difference

SAP Worksheet #28-5— QC Samples Table

Matrix:	Aqueous
Analytical Group:	TCLPV
Analytical Method / SOP Reference:	SW-846 1311, 8260B / H7, M5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Every 12 hours	No target analytes > Quantitation Limit Surrogates must be within: 1,2-Dichloroethane-d4: 70-120% 4-bromofluorobenzene: 75-120% 1,2-Dichlorobenzene-d4: 64-132% Toluene-d8: 85-120%	Re-clean and re-analyze	Nathan Krueger	Bias / Contamination	No target analytes > Quantitation Limit Surrogates must be within: 1,2-Dichloroethane-d4: 70-120% 4-bromofluorobenzene: 75-120% 1,2-Dichlorobenzene-d4: 64-132% Toluene-d8: 85-120%
Surrogates	Each sample	Surrogates must be within: 1,2-Dichloroethane-d4: 70-120% 4-bromofluorobenzene: 75-120% 1,2-Dichlorobenzene-d4: 64-132% Toluene-d8: 85-120%	Check instrument performance, re-analyze and qualify data	Nathan Krueger	Accuracy / Bias	Surrogates must be within: 1,2-Dichloroethane-d4: 70-120% 4-bromofluorobenzene: 75-120% 1,2-Dichlorobenzene-d4: 64-132% Toluene-d8: 85-120%
Laboratory Control Sample	1 per batch or 1 per 20 samples	Benzene: 80-120% Carbon tetrachloride: 65-140% Chlorobenzene: 80-120% Chloroform: 65-135% 1,2-Dichloroethane: 70-130% 1,1-Dichloroethene: 70-130% 2-Butanone: 30-150% Tetrachloroethene: 45-150% Trichloroethene: 70-125% Vinyl Chloride:50-145%	Check instrument performance, reanalyze	Nathan Krueger	Accuracy / Bias	Benzene: 80-120% Carbon tetrachloride: 65-140% Chlorobenzene: 80-120% Chloroform: 65-135% 1,2-Dichloroethane: 70-130% 1,1-Dichloroethene: 70-130% 2-Butanone: 30-150% Tetrachloroethene: 45-150% Trichloroethene: 70-125% Vinyl Chloride:50-145%
Laboratory Control Sample Duplicate	1 per batch or 1 per 20 samples if no MS/MSD	Same acceptance criteria as LCS	Check instrument performance, reanalyze	Nathan Krueger	Accuracy / Bias	Same acceptance criteria as LCS
Internal Standards	Each sample	Area counts –50% to +100% of Initial Calibration IS or Continuing Calibration IS area counts; Retention times +/- 30 secs of Continuing Calibration	Check instrument performance, reanalyze and qualify data	Nathan Krueger	Precision / Accuracy / Bias	Area counts –50% to +100% of Initial Calibration IS or Continuing Calibration IS area counts; Retention times +/- 30 secs of Continuing Calibration
Matrix spike/ Matrix spike duplicate	Every 20 samples	Same acceptance criteria as LCS	Check instrument performance, qualify data	Nathan Krueger	Precision / Accuracy / Bias	Same acceptance criteria as LCS

SAP Worksheet #28-6—QC Samples Table

Matrix:	Aqueous
Analytical Group:	TCLPS
Analytical Method / SOP Reference:	SW-846 1311, 8270C / H7, P5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch or 1 per 20 samples	No target analytes > Quantitation Limit Surrogates within: 2-Fluorobiphenyl: 46 -108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Nitrobenzene-d5: 38-122%	Re-clean and re-analyze	Hall Moore	Bias / Contamination	No target analytes > Quantitation Limit Surrogates within: 2-Fluorobiphenyl: 46 -108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Nitrobenzene-d5: 38-122%
Surrogates	Each sample	Surrogates within: 2-Fluorobiphenyl: 46 -108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Nitrobenzene-d5: 38-122%	Check instrument performance, re-analyze and qualify data	Hall Moore	Accuracy / Bias	Surrogates within: 2-Fluorobiphenyl: 46 -108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Nitrobenzene-d5: 38-122%
Laboratory Control Sample	1 per batch or 1 per 20 samples	2-methylphenol: 17-153% 3&4-methylphenol: 21-143% 1,4-Dichlorobenzene: 24-144% 2,4-Dinitrotoluene: 33-153% Hexachlorobenzene: 24-110% Hexachlorobutadiene: 25-137% Hexachloroethane: 23-147% Nitrobenzene: 23-147% Pentachlorophenol: 19-110% Pyridine: 23-121% 2,4,5-Trichlorphenol: 28-144% 2,4,6-Trichlorophenol: 31-147%	Check instrument performance, reanalyze	Hall Moore	Accuracy / Bias	2-methylphenol: 17-153% 3&4-methylphenol: 21-143% 1,4-Dichlorobenzene: 24-144% 2,4-Dinitrotoluene: 33-153% Hexachlorobenzene: 24-110% Hexachlorobutadiene: 25-137% Hexachloroethane: 23-147% Nitrobenzene: 23-147% Pentachlorophenol: 19-110% Pyridine: 23-121% 2,4,5-Trichlorphenol: 28-144% 2,4,6-Trichlorophenol: 31-147%
Laboratory Control Sample Duplicate	1 per batch or 1 per 20 samples if no MS/MSD	Same acceptance criteria as LCS	Check instrument performance, reanalyze	Hall Moore	Accuracy / Bias	Same acceptance criteria as LCS
Internal Standards	Each sample	Area counts –50% to +100% of Initial Calibration IS or Continuing Calibration IS area counts; Retention times +/- 30 secs of Continuing Calibration	Check instrument performance, reanalyze and qualify data	Hall Moore	Precision / Accuracy / Bias	Area counts –50% to +100% of Initial Calibration IS or Continuing Calibration IS area counts; Retention times +/- 30 secs of Continuing Calibration
Matrix spike/ Matrix spike duplicate	Every 20 samples	Same acceptance criteria as LCS	Check instrument performance, qualify data	Hall Moore	Precision / Accuracy / Bias	Same acceptance criteria as LCS

SAP Worksheet #28-7—QC Samples Table

Matrix:	Aqueous
Analytical Group:	TCLPP
Analytical Method / SOP Reference:	SW-846 1311, 8081A / H7, Q.6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch or 1 per 20 samples	No target analytes > Quantitation Limit surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%	Re-clean and re-analyze	Rekha Patel	Bias / Contamination	No target analytes > Quantitation Limit surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%
Surrogates	Each sample	surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%	Check instrument performance, reanalyze, qualify data	Rekha Patel	Accuracy / Bias	surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%
Laboratory Control Sample	1 per batch or 1 per 20 samples	Endrin: 43-134% Heptachlor: 45-128% Heptachlor epoxide: 53-134% Gamma-BHC: 73-125% Methoxychlor: 73-142%	Check instrument performance, reanalyze	Rekha Patel	Accuracy / Bias	Endrin: 43-134% Heptachlor: 45-128% Heptachlor epoxide: 53-134% Gamma-BHC: 73-125% Methoxychlor: 73-142%
Laboratory Control Sample Duplicate	1 per batch or 1 per 20 samples if no MS/MSD	Same acceptance criteria as LCS	Check instrument performance, reanalyze	Rekha Patel	Accuracy / Bias	Same acceptance criteria as LCS
Matrix spike / Matrix spike duplicate	Every 20 samples	Same acceptance criteria as LCS	Check instrument performance, qualify data	Rekha Patel	Precision / Accuracy / Bias	Same acceptance criteria as LCS

SAP Worksheet #28-8—QC Samples Table

Matrix:	Aqueous
Analytical Group:	TCLPM
Analytical Method / SOP Reference:	SW-846 1311, 6010B, 7470A / H.7, H.10, H.12

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch or 1 per 20 samples	No target analytes > ½ Quantitation Limit	Re-digest and reanalyze	Rita Amin	Bias / Contamination	No target analytes > ½ Quantitation Limit
Laboratory Control Sample	1 per batch or 1 per 20 samples	%Recovery 80% - 120%	Re-digest and reanalyze	Rita Amin	Accuracy / Bias / Contamination	%Recovery 80% - 120%
Duplicate Sample	1 per 20 samples	RPD <=20%	Qualify Data	Rita Amin	Precision	RPD <=20%
Matrix Spike	1 per 20 samples	%Recovery 80% - 120%	Perform post-digestion spike analysis, qualify data	Rita Amin	Accuracy / Bias	%Recovery 80% - 120%
Post-digestion Spike	For compounds outside of QC limits in Matrix Spike	%Recovery 75-125%	Qualify data	Rita Amin	Accuracy / Bias	%Recovery 75-125%
ICP Serial Dilution	Per analytical run	%Difference <10%	Qualify data	Rita Amin	Accuracy / Bias	%Difference <10%

SAP Worksheet #28-9—QC Samples Table

Matrix:	Aqueous
Analytical Group:	TCLPH
Analytical Method / SOP Reference:	SW-846 1311, 8151A / H7, Q.10

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch or 1 per 20 samples	No target analytes > Quantitation Limit surrogate values within lab statistical QC limits: DCAA: 61-136%	Re-clean and re-analyze	Rekha Patel	Bias / Contamination	No target analytes > Quantitation Limit surrogate values within lab statistical QC limits: DCAA: 61-136%
Surrogates	Each sample	surrogate values within lab statistical QC limits: DCAA: 61-136%	Check instrument performance, reanalyze, qualify data	Rekha Patel	Accuracy / Bias	surrogate values within lab statistical QC limits: DCAA: 61-136%
Laboratory Control Sample	1 per batch or 1 per 20 samples	2,4-D: 61-136% 2,4,5-TP: 61-136%	Check instrument performance, reanalyze	Rekha Patel	Accuracy / Bias	2,4-D: 61-136% 2,4,5-TP: 61-136%
Laboratory Control Sample Duplicate	1 per batch or 1 per 20 samples if no MS/MSD	Same acceptance criteria as LCS	Check instrument performance, reanalyze	Rekha Patel	Accuracy / Bias	Same acceptance criteria as LCS
Matrix spike / Matrix spike duplicate	Every 20 samples	Same acceptance criteria as LCS	Check instrument performance, qualify data	Rekha Patel	Precision / Accuracy / Bias	Same acceptance criteria as LCS

SAP Worksheet #28-10—QC Samples Table

Matrix:	Aqueous
Analytical Group:	CORR
Analytical Method / SOP Reference:	SW-846 9045 / J.12

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Lab control sample (pH 7.0 buffer)	Every 10 samples	+/- 0.10 pH units	Recalibrate/reanalyze	James Anderson	Accuracy, Precision	+/- 0.10 pH units
Duplicate	One set per 20 field samples	Relative Percent Difference <=15%	Qualify Data	James Anderson	Precision	Relative Percent Difference <=15%

SAP Worksheet #28-11—QC Samples Table

Matrix:	Aqueous
Analytical Group:	IGN
Analytical Method / SOP Reference:	Pensky Martens / N.1

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Lab Control Sample	One per batch of 20 or fewer samples	%Recovery 80% - 120%	Reanalyze	Namory Keita	Accuracy	%Recovery 80% - 120%
Duplicate	One set per 20 field samples, for every sample that flashes, or extinguishes flame <140 degrees	Relative Percent Difference <=20%	Repeat, Qualify Data	Namory Keita	Precision	Relative Percent Difference <=20%

SAP Worksheet #28-12—Laboratory QC Samples Table

Matrix:	Solid
Analytical Group:	TCLPV
Analytical Method / SOP Reference:	SW-846 1311, 8260B / H7, M.5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Every 12 hours	No target analytes > Quantitation Limit Surrogates must be within: 1,2-Dichloroethane-d4: 70-120% 4-bromofluorobenzene: 75-120% 1,2-Dichlorobenzene-d4: 64-132% Toluene-d8: 85-120%	Re-clean and re-analyze	Nathan Krueger	Bias / Contamination	No target analytes > Quantitation Limit Surrogates must be within: 1,2-Dichloroethane-d4: 70-120% 4-bromofluorobenzene: 75-120% 1,2-Dichlorobenzene-d4: 64-132% Toluene-d8: 85-120%
Surrogates	Each sample	Surrogates must be within: 1,2-Dichloroethane-d4: 70-120% 4-bromofluorobenzene: 75-120% 1,2-Dichlorobenzene-d4: 64-132% Toluene-d8: 85-120%	Check instrument performance, re-analyze and qualify data	Nathan Krueger	Accuracy / Bias	Surrogates must be within: 1,2-Dichloroethane-d4: 70-120% 4-bromofluorobenzene: 75-120% 1,2-Dichlorobenzene-d4: 64-132% Toluene-d8: 85-120%
Laboratory Control Sample	1 per batch or 1 per 20 samples	Benzene: 80-120% Carbon tetrachloride: 65-140% Chlorobenzene: 80-120% Chloroform: 65-135% 1,2-Dichloroethane: 70-130% 1,1-Dichloroethene: 70-130% 2-Butanone: 30-150% Tetrachloroethene: 45-150% Trichloroethene: 70-125% Vinyl Chloride:50-145%	Check instrument performance, reanalyze	Nathan Krueger	Accuracy / Bias	Benzene: 80-120% Carbon tetrachloride: 65-140% Chlorobenzene: 80-120% Chloroform: 65-135% 1,2-Dichloroethane: 70-130% 1,1-Dichloroethene: 70-130% 2-Butanone: 30-150% Tetrachloroethene: 45-150% Trichloroethene: 70-125% Vinyl Chloride:50-145%
Laboratory Control Sample Duplicate	1 per batch or 1 per 20 samples if no MS/MSD	Same acceptance criteria as LCS	Check instrument performance, reanalyze	Nathan Krueger	Accuracy / Bias	Same acceptance criteria as LCS
Internal Standards	Each sample	Area counts –50% to +100% of Initial Calibration IS or Continuing Calibration IS area counts; Retention times +/- 30 secs of Continuing Calibration	Check instrument performance, reanalyze and qualify data	Nathan Krueger	Precision / Accuracy / Bias	Area counts –50% to +100% of Initial Calibration IS or Continuing Calibration IS area counts; Retention times +/- 30 secs of Continuing Calibration
Matrix spike/ Matrix spike duplicate	Every 20 samples	Same acceptance criteria as LCS	Check instrument performance, qualify data	Nathan Krueger	Precision / Accuracy / Bias	Same acceptance criteria as LCS

SAP Worksheet #28-13—Laboratory QC Samples Table

Matrix:	Solid
Analytical Group:	TCLPS
Analytical Method / SOP Reference:	SW-846 1311, 8270C / H7, P.5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch or 1 per 20 samples	No target analytes > Quantitation Limit Surrogates within: 2-Fluorobiphenyl: 46 -108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Nitrobenzene-d5: 38-122%	Re-clean and re-analyze	Hall Moore	Bias / Contamination	No target analytes > Quantitation Limit Surrogates within: 2-Fluorobiphenyl: 46 -108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Nitrobenzene-d5: 38-122%
Surrogates	Each sample	Surrogates within: 2-Fluorobiphenyl: 46 -108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Nitrobenzene-d5: 38-122%	Check instrument performance, re-analyze and qualify data	Hall Moore	Accuracy / Bias	Surrogates within: 2-Fluorobiphenyl: 46 -108% Terphenyl-d14: 29-133% 2,4,6-Tribromophenol: 35-157% 2-Fluorophenol: 28-116% Nitrobenzene-d5: 38-122%
Laboratory Control Sample	1 per batch or 1 per 20 samples	2-methylphenol: 17-153% 3&4-methylphenol: 21-143% 1,4-Dichlorobenzene: 24-144% 2,4-Dinitrotoluene: 33-153% Hexachlorobenzene: 24-110% Hexachlorobutadiene: 25-137% Hexachloroethane: 23-147% Nitrobenzene: 23-147% Pentachlorophenol: 19-110% Pyridine: 23-121% 2,4,5-Trichlorphenol: 28-144% 2,4,6-Trichlorophenol: 31-147%	Check instrument performance, reanalyze	Hall Moore	Accuracy / Bias	2-methylphenol: 17-153% 3&4-methylphenol: 21-143% 1,4-Dichlorobenzene: 24-144% 2,4-Dinitrotoluene: 33-153% Hexachlorobenzene: 24-110% Hexachlorobutadiene: 25-137% Hexachloroethane: 23-147% Nitrobenzene: 23-147% Pentachlorophenol: 19-110% Pyridine: 23-121% 2,4,5-Trichlorphenol: 28-144% 2,4,6-Trichlorophenol: 31-147%
Laboratory Control Sample Duplicate	1 per batch or 1 per 20 samples if no MS/MSD	Use acceptance criteria from LCS	Check instrument performance, reanalyze	Laboratory Supervisor	Accuracy / Bias	Use acceptance criteria from LCS
Internal Standards	Each sample	Area counts –50% to +100% of Initial Calibration IS or Continuing Calibration IS area counts; Retention times +/- 30 secs of Continuing Calibration	Check instrument performance, reanalyze and qualify data	Laboratory Supervisor	Precision / Accuracy / Bias	Area counts –50% to +100% of Initial Calibration IS or Continuing Calibration IS area counts; Retention times +/- 30 secs of Continuing Calibration
Matrix spike/ Matrix spike duplicate	Every 20 samples	Use acceptance criteria from LCS	Check instrument performance, qualify data	Laboratory Supervisor	Precision / Accuracy / Bias	Use acceptance criteria from LCS

SAP Worksheet #28-14—Laboratory QC Samples Table

Matrix:	Solid
Analytical Group:	TCLPP
Analytical Method / SOP Reference:	SW-846 1311, 8081A / H7, Q.6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch or 1 per 20 samples	No target analytes > Quantitation Limit surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%	Re-clean and re-analyze	Rekha Patel	Bias / Contamination	No target analytes > Quantitation Limit surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%
Surrogates	Each sample	surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%	Check instrument performance, reanalyze, qualify data	Rekha Patel	Accuracy / Bias	surrogates within: Decachlorobiphenyl: 16-166% TCMX: 6-154%
Laboratory Control Sample	1 per batch or 1 per 20 samples	Endrin: 43-134% Heptachlor: 45-128% Heptachlor epoxide: 53-134% Gamma-BHC: 73-125% Methoxychlor: 73-142%	Check instrument performance, reanalyze	Rekha Patel	Accuracy / Bias	Endrin: 43-134% Heptachlor: 45-128% Heptachlor epoxide: 53-134% Gamma-BHC: 73-125% Methoxychlor: 73-142%
Laboratory Control Sample Duplicate	1 per batch or 1 per 20 samples if no MS/MSD	Same acceptance criteria as LCS	Check instrument performance, reanalyze	Rekha Patel	Accuracy / Bias	Same acceptance criteria as LCS
Matrix spike / Matrix spike duplicate	Every 20 samples	Same acceptance criteria as LCS	Check instrument performance, qualify data	Rekha Patel	Precision / Accuracy / Bias	Same acceptance criteria as LCS

SAP Worksheet #28-15—Laboratory QC Samples Table

Matrix:	Solid
Analytical Group:	TCLPH
Analytical Method / SOP Reference:	SW-846 1311, 8151A / H7, Q.10

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch or 1 per 20 samples	No target analytes > Quantitation Limit surrogate values within lab statistical QC limits: DCAA: 61-136%	Re-clean and re-analyze	Rekha Patel	Bias / Contamination	No target analytes > Quantitation Limit surrogate values within lab statistical QC limits: DCAA: 61-136%
Surrogates	Each sample	surrogate values within lab statistical QC limits: DCAA: 61-136%	Check instrument performance, reanalyze, qualify data	Rekha Patel	Accuracy / Bias	surrogate values within lab statistical QC limits: DCAA: 61-136%
Laboratory Control Sample	1 per batch or 1 per 20 samples	2,4-D: 61-136% 2,4,5-TP: 61-136%	Check instrument performance, reanalyze	Rekha Patel	Accuracy / Bias	2,4-D: 61-136% 2,4,5-TP: 61-136%
Laboratory Control Sample Duplicate	1 per batch or 1 per 20 samples if no MS/MSD	Same acceptance criteria as LCS	Check instrument performance, reanalyze	Rekha Patel	Accuracy / Bias	Same acceptance criteria as LCS
Matrix spike / Matrix spike duplicate	Every 20 samples	Same acceptance criteria as LCS	Check instrument performance, qualify data	Rekha Patel	Precision / Accuracy / Bias	Same acceptance criteria as LCS

SAP Worksheet #28-16—Laboratory QC Samples Table

Matrix:	Solid
Analytical Group:	TCLPM
Analytical Method / SOP Reference:	SW-846 1311, 6010B, 7470A / H.7, H.10, H.12

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch or 1 per 20 samples	No target analytes > ½ Quantitation Limit	Re-digest and reanalyze	Rita Amin	Bias / Contamination	No target analytes > ½ Quantitation Limit
Laboratory Control Sample	1 per batch or 1 per 20 samples	%Recovery 80% - 120%	Re-digest and reanalyze	Rita Amin	Accuracy / Bias / Contamination	%Recovery 80% - 120%
Duplicate Sample	1 per 20 samples	RPD <=20%	Qualify Data	Rita Amin	Precision	RPD <=20%
Matrix Spike	1 per 20 samples	%Recovery 80% - 120%	Perform post-digestion spike analysis, qualify data	Rita Amin	Accuracy / Bias	%Recovery 80% - 120%
Post-digestion Spike	For compounds outside of QC limits in Matrix Spike	%Recovery 75-125%	Qualify data	Rita Amin	Accuracy / Bias	%Recovery 75-125%
ICP Serial Dilution	Per analytical run	%Difference <10%	Qualify data	Rita Amin	Accuracy / Bias	%Difference <10%

SAP Worksheet #28-17—Laboratory QC Samples Table

Matrix:	Solid
Analytical Group:	CORR
Analytical Method / SOP Reference:	SW-846 9045 / J12

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Lab control sample (pH 7.0 buffer)	Every 10 samples	+/- 0.10 pH units	Recalibrate/reanalyze	James Anderson	Accuracy, Precision	+/- 0.10 pH units
Duplicate	One set per 20 field samples	Relative Percent Difference <=15%	Qualify Data	James Anderson	Precision	Relative Percent Difference <=15%

SAP Worksheet #28-18—Laboratory QC Samples Table

Matrix:	Solid
Analytical Group:	IGN
Analytical Method / SOP Reference:	Pensky Martens / N.1

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Lab Control Sample	One per batch of 20 or fewer samples	%Recovery 80% - 120%	Reanalyze	Namory Keita	Accuracy	%Recovery 80% - 120%
Duplicate	One set per 20 field samples, for every sample that flashes, or extinguishes flame <140 degrees	Relative Percent Difference <=20%	Repeat, Qualify Data	Namory Keita	Precision	Relative Percent Difference <=20%

SAP Worksheet #29—Project Documents and Records Table

Document	Where Maintained
• Field Notebooks • Chain-of-Custody Records • Air Bills • Custody Seals • Corrective Action Forms • Electronic Data Deliverables • Identification of QC Samples • Meteorological Data from Field • Sampling instrument calibration logs • Sampling locations and sampling plan • Sampling notes and drilling logs • Water quality parameters • Sample Receipt, Chain-of-Custody, and Tracking Records • Standard Traceability Logs • Equipment Calibration Logs • Sample Prep Logs • Run Logs • Equipment Maintenance, Testing, and Inspection Logs • Corrective Action Forms • Reported Field Sample Results • Reported Result for Standards, QC Checks, and QC Samples • Instrument printouts (raw data) for Field Samples, Standards, QC Checks, and QC Samples • Data Package Completeness Checklists • Sample disposal records • Extraction/Clean-up Records • Raw Data (stored on disk) • Fixed Laboratory Audit Checklists • Data Validation Reports • Corrective Action Forms • Laboratory QA Plan • MDL Study Information • IDW Disposal Chit	• Field data deliverables such as logbooks entries, chain of custodies, air bills, EDDs, etc will be kept on CH2M HILLCH2M HILL's local internet server. • Field parameter data will be loaded with the analytical data into EnDat • Analytical laboratory hardcopy deliverables and data validation reports will be saved on the network server. • Electronic data from the laboratory will be loaded into EnDat

SAP Worksheet #30—Analytical Services Table

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory/ Organization	Backup Laboratory/ Organization
Aqueous	Full TCLP, Corrosivity, Ignitability	See Figure 3 and Worksheet 18	SW846 1311/ 8260B, 8270C, 8081A, 8151A, 6010B, 7470A/7471A, SW-846 9045C, Pensky Martens	28 days	GPL Laboratories, LLLP 7210A Corporate Court, Frederick, MD 21703 301-694-5310 Virginia Zusman	TBD
Solid						
GW	Pesticides		SW846 8081A			
	PCBs		SW846 8082			
SS	Pesticides		SW846 8081A			
	PCBs		SW846 8082			

All samples will be delivered to the off-site analytical laboratory, GPL Laboratories.

A backup laboratory is not determined at this time. One will be procured in the event that GPL Laboratories is no longer able to provide analytical services.

SAP Worksheet #31—Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA)	Person(s) Responsible for Monitoring Effectiveness of CA
Offsite Laboratory Technical Systems Audit	Laboratory must have current Naval Facilities Engineering Service Center (NFESC) evaluation letter which will identify the period of performance. The laboratory must be re-evaluated prior to expiration of period of performance	External	U.S. Navy (NFESC)	Project QA Officer- Pati Moreno/ NFESC, Port Hueneme, CA	Subcontracted Laboratory's QA Officer	Subcontracted Laboratory's QA Officer	Project QA Officer- Anita Dodson- CH2M HILL

SAP Worksheet #32—Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Laboratory Performance and Systems Audits	Written Audit Report	Elsa Tai. GPL QA Manager	Within 2 months of audit	Memorandum	NFESC Auditor, TBD	Within two months of receipt of initial notification.

An example Corrective Action Form is included in Appendix B.

SAP Worksheet #33—QA Management Reports Table

Type of Report	Frequency	Projected Delivery Date	Person Responsible for Report Preparation	Report Recipient(s)
Site Investigation Report	Post- Field Event	November 2008	Bill Hannah/CH2M HILL	Stakeholders, see Worksheet 4

The Site Investigation Report will address the following:

- Summary of project QA/QC requirements/procedures
- Conformance of project to UFP-SAP requirements/procedures
- Status of project schedule
- Deviations from the UFP-SAP and approved amendments that were made
- Results of data review activities (how much usable data was generated)
- Corrective actions if needed and their effectiveness
- Data usability with regards to: precision, accuracy, representativeness, completeness, comparability, and sensitivity
- Limitations on data use

SAP Worksheet #34—Verification (Step I) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Planning Documents	Evidence of approval and completeness of UFP-SAP.	Internal	Bill Hannah CH2M HILL
Chain of Custody and shipping forms	CoC forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the CoC will be initialed by the reviewer, a copy of the CoC retained in the site file, and the original and remaining copies taped inside the cooler for shipment. See CoC SOP (on CD) for further details.	Internal	FTL and Genevieve Moore CH2M HILL
Field Log Notebooks	Field notes will be reviewed to ensure completeness of field data parameters, shipping information, and sample collection times, etc. The logbook will also be used to document, explain, and justify all deviations from the approved work plan and UFP-SAP.	Internal	FTL CH2M HILL
Sample Receipt and Management	Upon their arrival at the laboratory, the samples will be cross-referenced against the COC records. All sample labels will be checked against the COC, and any mislabeling will be identified, investigated, and corrected. The samples will be logged in at every storage area and work station required by the designated analyses. Individual analysts will verify the completeness and accuracy of the data recorded on the forms.	Internal	GPL Laboratories employees
QC Summary Report	A summary of all QC sample results will be verified for completeness once the data is received from the laboratory.	External	Genevieve Moore CH2M HILL
Field Investigation Interpretive Data	Immediately following receipt of the analytical data from the laboratory and prior to submittal to the data validator, a population to population comparison will be conducted comparing site results and the results from the background sample set. The background population to population comparison for will be used to determine the likelihood of a release relative to background. The data will also be compared to screening criteria (see Worksheet 15).	Internal	Bill Hannah, Roni Warren, Dan Lavoie CH2M HILL

SAP Worksheet #35—Validation (Steps IIa and IIb) Process Table

Step IIa / IIb1	Validation Input	Description	Responsible for Validation
IIa	SOPs	Review field logbooks, laboratory case narratives, data deliverables for compliance to methods and signatures.	**FTL, Bill Hannah** *CH2M HILL*
IIa	QC Results	Establish that all QC samples were run and compliant with method-required limits as specified in Worksheet 12.	**Laura Maschhoff** *DataQual Environmental Services*
IIb	QC Results	Verify that QC samples were run and compliant with limits established in the UFP-SAP.	**Anita Dodson** *CH2M HILL* **Laura Maschhoff** *DataQual Environmental Services*
IIb	Project Quantification Limits	Ensure all sample results met the project quantification and action limits specified in Worksheet 15.	**Bill Hannah, Megan Hilton** *CH2M HILL*
IIb	Raw data	10% review of raw data to confirm laboratory calculations.	**Laura Maschhoff** *DataQual Environmental Services*

[1] IIa=compliance with methods, procedures, and contracts

IIb=comparison with measurement performance criteria in the SAP

SAP Worksheet #36—Analytical Data Validation (Steps IIa and IIb) Summary Table

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator
IIa	GW	Pesticides, PCBs	Analytical methods and laboratory SOPs as presented in this SAP will be used to evaluate compliance against QA/QC criteria. Should adherence to QA/QC criteria yield deficiencies, data may be qualified. The data qualifiers that may be used are those presented in *Region III Modification to National Functional Guidelines for Organic Data Review, MultiMedia, Multi Concentration (September 1994)*	**Laura Maschhoff** *DataQual Environmental Services*
IIa	SS			
IIa & b	Aqueous, Solid	Full TCLP, Corrosivity, Ignitability	Comparison to Project Action Limits to characterize the waste and method performance criteria	Anita Dodson, Bill Hannah, CH2M HILL
IIb	GW	Pesticides, PCBs	Comparison to Project Action Limits and method performance criteria	
IIb	SS			

SAP Worksheet #37—Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:

Non-detected contaminants will be evaluated to ensure that project required quantitation limits in Worksheet #15 were achieved. If project quantitation limits were achieved and the verification and validation steps yielded acceptable data, then the data is considered usable.

During verification and validation steps, data may be qualified as estimated with the following qualifiers: J, UJ, K, L, or UL. These qualifiers represent minor QC deficiencies which will not affect the usability of the data. When major QC deficiencies are encountered, data will be qualified with an R and in most cases is not considered usable for project decisions.

For statistical comparisons non-detect values will be represented by a concentration equal to one-half the sample reporting limit. For duplicate sample results, the most conservative value will be used for project decisions.

Analytical data will be checked to ensure the values and any qualifiers are appropriately transferred to the electronic database. These checks include comparison of hardcopy data and qualifiers to the electronic data deliverable. Once the data has been uploaded into the electronic database, another check will be performed to ensure all results were loaded accurately.

Field and laboratory precision will be compared as relative percent difference (RPD) between the two results.

Deviations from the SAP will be reviewed to assess whether corrective action is warranted and to assess impacts to achievement of project objectives.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

To assess whether a sufficient quantity of acceptable data are available for decision making, the data will be reconciled with measurement performance criteria following validation and review of data quality indicator.

If significant biases are detected with laboratory QA/QC samples it will be evaluated to assess impact on decision making. Low biases will be described in greater detail as they represent a possible inability to detect compounds that may be present at the site.

If significant deviations are noted between lab and field precision the cause will be further evaluated to assess impact on decision making.

Identify the personnel responsible for performing the usability assessment:

Megan Hilton, CH2M HILL; Bill Hannah, CH2M HILL

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

Data tables will be produced to reflect detected and non-detected site COC's and geochemical parameters. Data qualifiers will be reflected in the tables and discussed in the data quality evaluation.

A data quality evaluation will be provided as part of the technical memorandum prepared to assess remedy effectiveness.

The technical memorandum will identify any data usability limitations and make recommendations for corrective action if necessary.

Figures

\\aphrodite\proj\USNavFacEngCom\CherryPoint\Projects\OU1\Additional_Investigation\Sites15_17_40\Figure 1 - Base Location Map.mxd

Legend
- Military Installation
- County Boundary
- Rivers and Streams

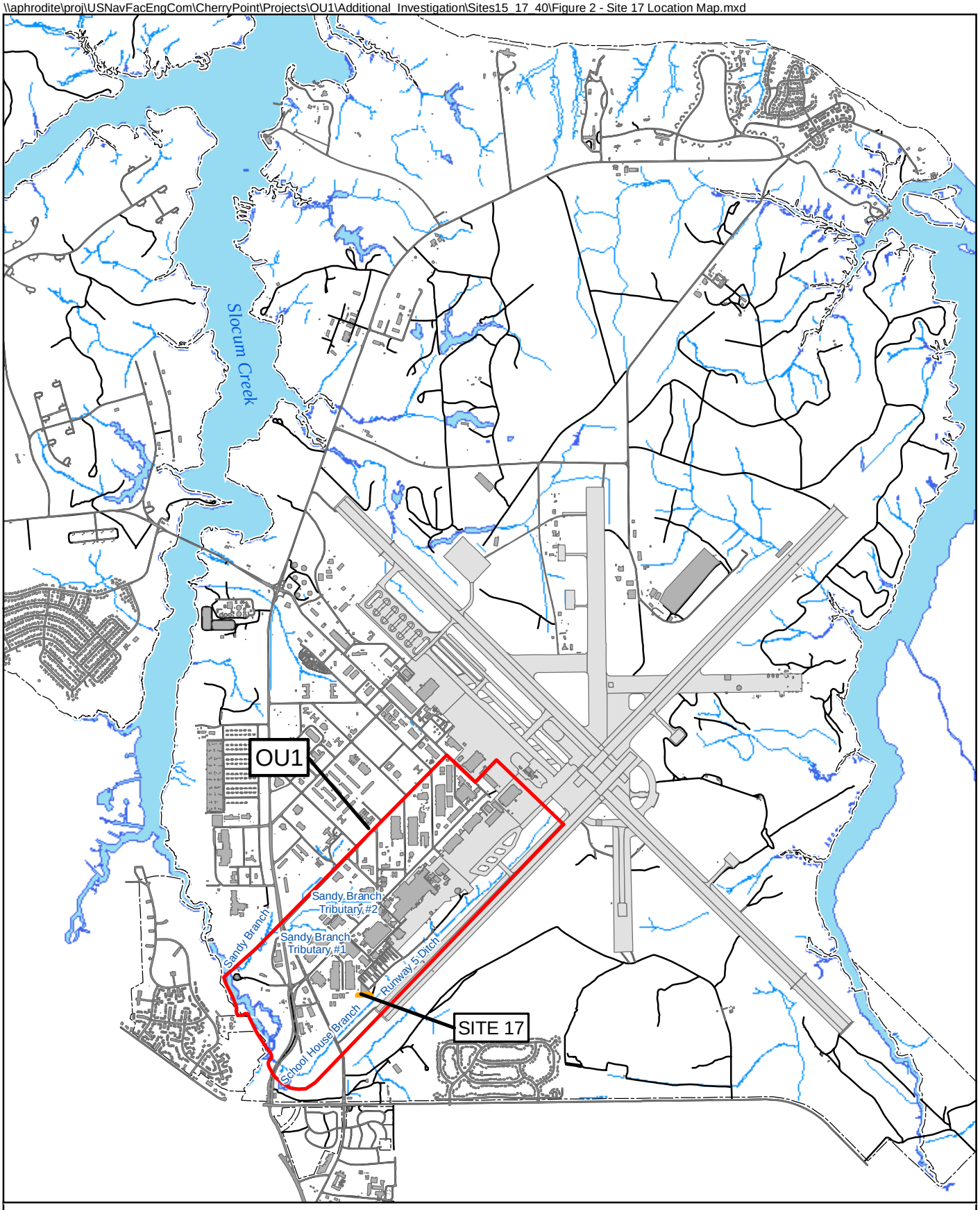
N
W E
S

0 20,000 40,000
Feet

Figure 1
Base Location Map
MCAS Cherry Point
North Carolina

CH2MHILL

\\aphrodite\proj\USNavFacEngCom\CherryPoint\Projects\OU1\Additional_Investigation\Sites15_17_40\Figure 2 - Site 17 Location Map.mxd



Legend

- OU Boundary
- Site Boundary
- Base Boundary
- Buildings
- Runway
- Road
- Surface Water

N
W E
S

0 1,500 3,000
Feet

Figure 2
Site 17 Location Map
MCAS Cherry Point
North Carolina

CH2MHILL

\\aphrodite\proj\USNavFaceNGCom\CherryPoint\Projects\OU1\Additional_Investigation\Figure 3 - Sites 17 Proposed Sampling Locations.mxd

Legend

- Proposed New Sampling Location (Dieldrin)
- Proposed New Sampling Location (PCBs)
- Monitoring Well
- Previous Soil Sampling Location
- Surface Water
- Excavated and Filled Areas (March, 1995)
- Site Boundary
- Active Railroad Line

ppm - parts per million
ppb - parts per billion
0.91 - Detected Total Aroclor Concentration in soil in ppm
PCB - Polychlorinated Biphenyl

N
W E
S

0 30 60
Feet

Figure 3
Proposed Sampling Locations at Site 17
MCAS Cherry Point
North Carolina

CH2MHILL

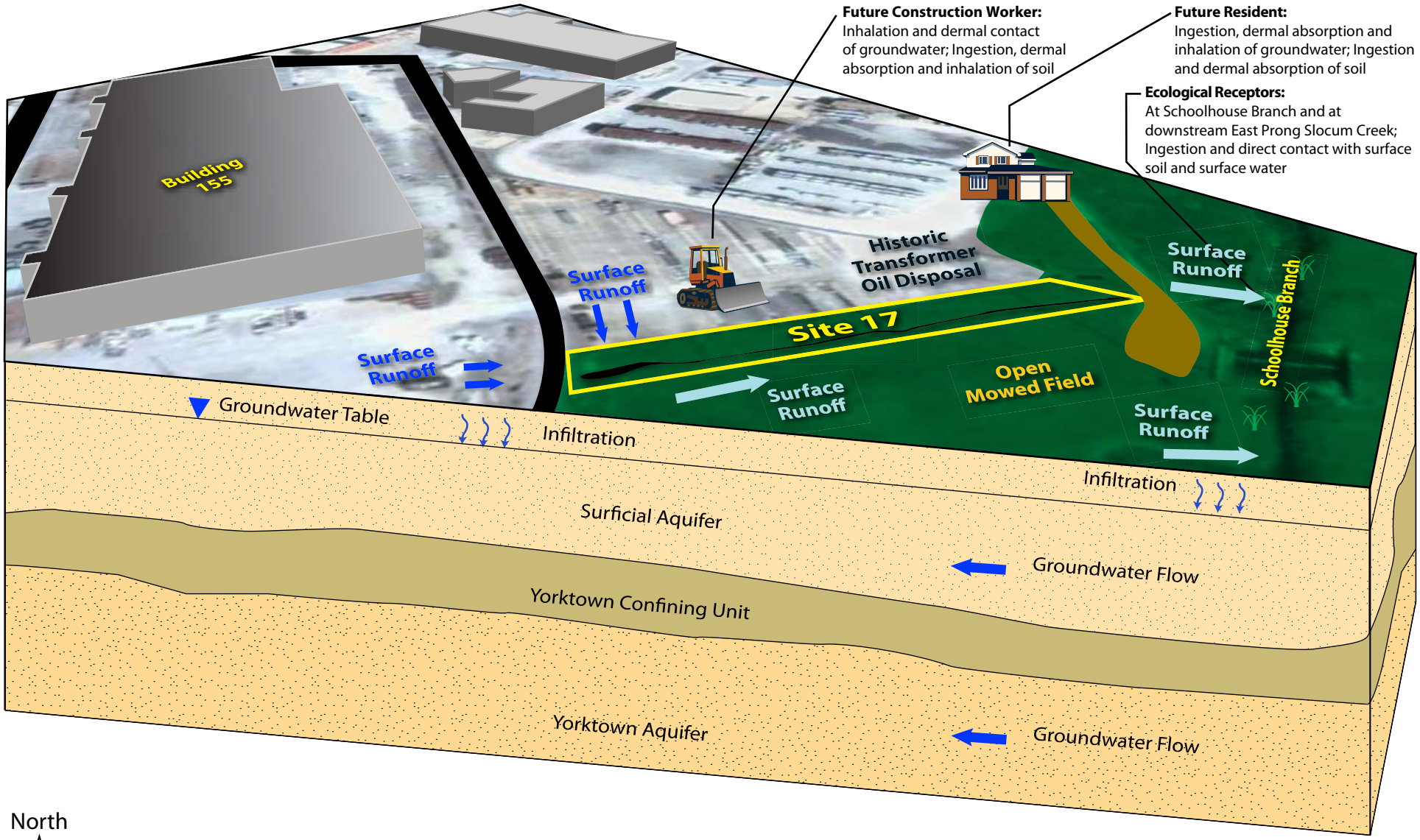
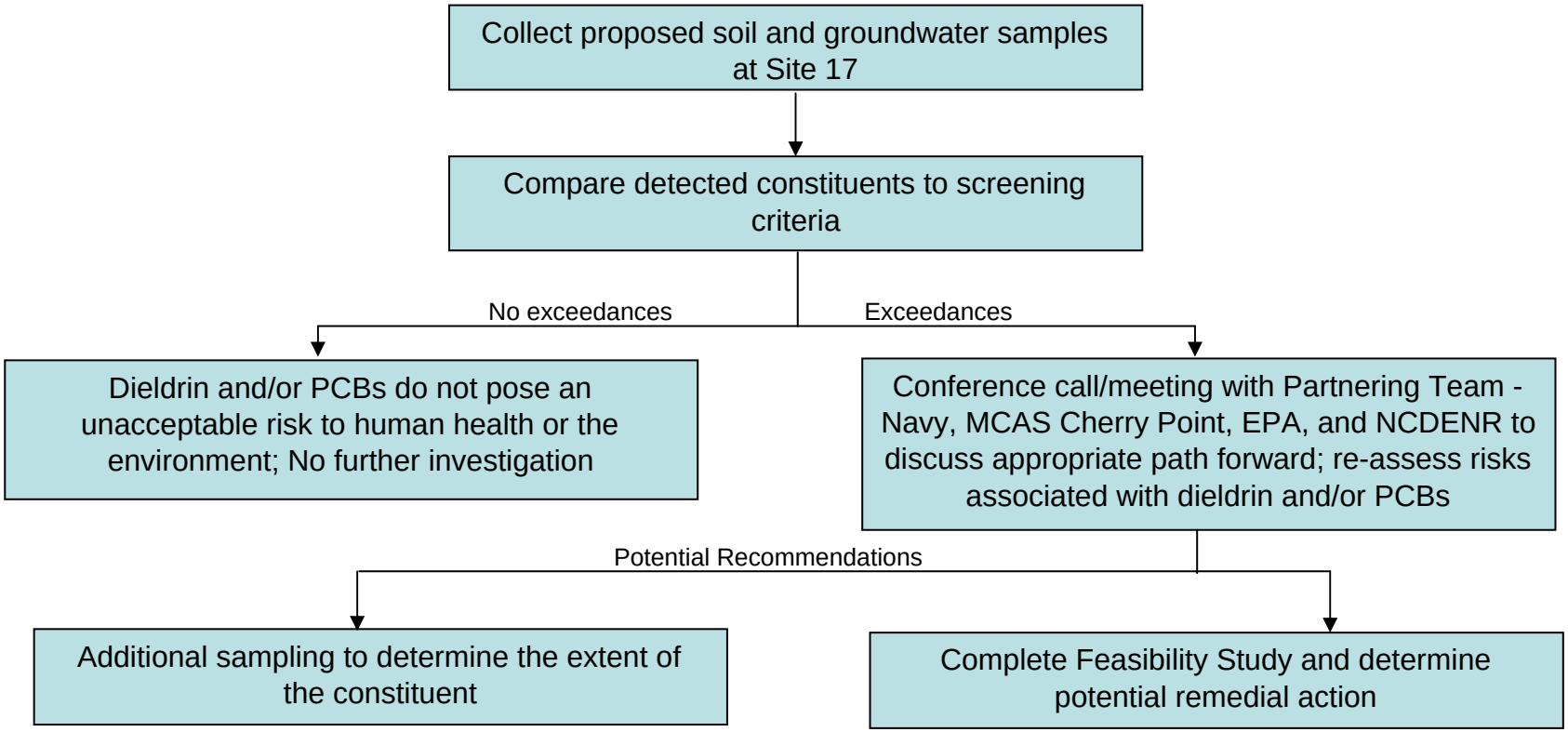


FIGURE 4
Simplified Conceptual Site Model of Site 17
Marine Corps Air Station Cherry Point
Cherry Point, North Carolina

CH2MHILL



PCBs - polychlorinated biphenyls
MCAS - Marine Corps Air Station
EPA - United States Environmental Protection Agency
NCDENR - North Carolina Department of Environment and Natural Resources

Figure 5
Decision Tree
Site 17
MCAS Cherry Point, North Carolina

Appendix A
Laboratory SOPs

SOP No: F.2

Title: Sample Receipt, Inspection, Preservation and Storage Condition Requirements

Scope: This Standard Operating Procedure describes procedures to be used by Sample Control personnel in the inspection of incoming samples and the preservation and storage requirements of those samples.

1.0 SAFETY CONSIDERATIONS

1.1 When working with laboratory samples, safety must be of prime concern. It is required that safety glasses, a lab coat, and gloves be worn while unpacking or handling samples. Broken samples should be treated as a chemical spill and should be managed accordingly, using proper spill cleanup procedures (see SOP "Spill Cleanup"). Bench tops should routinely be covered with a bench liner, which should be changed as needed. Good personal hygiene procedures should be followed while in the Sample Control Area, specifically, it is recommended that hands be washed before or immediately after leaving the area, food or drink shall not be consumed, cosmetics shall not be applied, and smoking is forbidden in the Sample Control Area and throughout all areas of the building.

1.2 Sample shipping containers must be placed under the fume hood prior to opening. Once the shipping container is opened, the contents should be examined for hazards prior to removal from the hood. If no obvious hazards are identified, the container may be removed from the hood for unpacking.

1.3 If samples are from DOE site of known or suspected radioactive hazard, the sample shipping containers must be screened for radioactivity prior to opening.

2.0 DOCUMENTATION OF INSPECTION

2.1 The inspection of all incoming sample shipments shall be documented on the Sample Receipt Checklist (Figure 1), except that those samples received under the Contract Laboratory Program shall be documented on CLP Form DC-1 (Figure 2). The Checklists shall be used to coordinate the inspection of the shipment and to record important information about the condition of the shipment and the documentation received.

2.2 The Sample Receipt Checklist documents the following information: The top section of the page documents receipt of the shipment, the identity of the client, the assigned Work Order Number and information relevant to the client's project. The next section of the checklist section contains specific items which must be checked and documented. Included in the section are spaces to document the sample and sample bottle count for comparison to the client supplied paper work and to document the necessity of a pH check, when applicable. If a pH check is required, it shall be documented on the Sample Preservation Check Documentation Form (Figure 3).

2.3 The final section for comments is used to explain any problems or discrepancies with the shipment. Any "no" response to the previous section must be documented in the spaces provided. The sample control personnel will sign and date the form as indicated at the bottom of the page.

2.4 CLP Form DC-1 is used in the same manner as the Sample Receipt Checklist and shall be used in place of that form for the documentation of inspection of CLP sample shipments. All CLP samples received must be documented individually on Form DC-1.

2.5 The completed checklist is included with the original paper work submitted to the Project Management Group to be placed in the central project file. A copy of the completed Sample Receipt Checklist (or Form DC-1) is filed with copies of client supplied paper work, the Work Order and Chain-of-Custody documentation in the Sample Log book maintained by Sample Control.

2.6 Documentation of any problems or discrepancies associated with sample receipt is performed by the Sample Coordinator on the Sample Receipt Checklist (or Form DC-1). The Project Management Group is then informed of these items and is responsible in resolving the issues with the client. Resolution to any problems must be documented by the Project Management Group.

3.0 SAMPLE SHIPMENT RECEIPT

3.1 Each shipment of samples arriving by commercial carrier must be accompanied by an airbill, airbill sticker, or manifest. The Sample Coordinator (also referred to as the Sample Custodian for EPA CLP work), upon taking custody of the shipment, shall sign the airbill/manifest and record the date and time the shipment was received in the space provided on the document. If no space is provided, documentation may be recorded on any open space or the back of the document.

3.2 The Sample Coordinator shall next initiate a Sample Receipt Checklist or in the case of a CLP shipment, Form DC-1. The samples must be accompanied with a Chain of Custody (COC) document. The sample acceptance policy must include that:

A. The person submitting the sample must provide full documentation with the sample, which must include:

- Sample Identification
- The location, date and time of collection
- The collector name, preservative added
- Matrix
- Any special remarks concerning the sample

B. Each sample or group of samples must include trip blanks, field blanks, equipment blanks, duplicates or other field submitted quality measures as required by the method.

C. Each sample must show evidence of proper preservation and use of sample containers allowed by the test method.

D. Each sample must be of adequate volume for the requested testing.

Any deviation from the policy, the client will be contacted and documented. The sample(s) will not be processed.

Where required by method, pH of the samples must be determined and reported on the checklist. The client name, date and time received, the carrier name, and the name of the Sample Coordinator shall be recorded in the appropriate sections of the forms. The presence or absence of the airbill/manifest shall be documented and, if present, the airbill/manifest number shall be recorded. Then, the temperature blank vial should be located, and the cooler temperature must be obtained, by inserting the calibrated thermometer into the temp. blank vial, and reading the temp. after 60-90 seconds. If no temp. blank exists, then the thermometer must be inserted in the center of the cooler, where the samples are located, and the temp. should be read after 60-90 seconds of thermometer insertion.

3.3 In the event that samples must be received on a weekend or holiday, special arrangements between the client and the laboratory must be established. Generally, clients must contact the Project Management Group to arrange for the special service. At that time Client Services shall ascertain if any samples requiring immediate analysis are to be included in the shipment. If this is the case, arrangements shall be made to have analytical staff available to perform the required analyses. Project Management Group shall arrange to have a person from Sample Control, or a properly trained alternate, available to receive the shipment. The shipment shall be received and inspected as per this procedure. The appropriate paperwork shall be completed and, if necessary, sample preservation shall be verified. Samples requiring immediate analysis are released upon receipt to the lab by the sample control personnel, before the completion of the transmission of the work order. Thereby enabling the laboratory to meet the required holding time criteria.

4.0 SHIPPING CONTAINER INSPECTION

4.1 All sample shipping containers shall be inspected for integrity and physical damage upon receipt. This shall be documented on Line 2 of the Checklist. Any problems found must be documented in the comments section at the bottom of the page. For Form DC-1, comments shall be made in the Remarks section.

4.2 Safety Note: If a strong smell is emanating from a shipping container, place the container in the hood to be unpacked. If a liquid is leaking from a shipping container, place container in the hood on a metal or plastic tray to contain the liquid and prevent gross contamination of the Sample Control Area. Any leaking material should be considered as a hazardous material and should be treated as such. All sample shipments originating from the Sample Management Office (SMO) for the Contract Laboratory Program (CLP) must be opened and unpacked under the hood. In all cases, proceed with caution while checking for broken sample vessels.

4.3 Once the shipping container has been examined for damage, and the presence or absence of custody seals is determined. The sample receiving personnel will evaluate and document the use of chain of custody seals that are of the non-tamper evident variety. Then the overall condition should be ascertained. This check shall be documented in the appropriate spaces on the forms.

5.0 SAMPLE VESSEL INSPECTION

5.1 The shipping container shall be carefully opened, any paper work should be removed, and it shall be determined whether ice or cold packs are present. The temperature of the shipping container shall be measured by placing a thermometer in the provided temp blank or among the samples, closing the lid and reopening the container and reading the thermometer for approximately 60-90 seconds. On the sample receipt checklist, document the presence or absence of ice or cold packs and the temperature of the shipping container. Please note that if the samples do not require cold preservation, for example preserved water samples for metals analysis, "No" must be checked and an explanation of N/A included in the Comments section.

5.2 The shipping container should be carefully unpacked and the incoming samples checked for:

- Physical damage due to inadequate packing and/or protection.
- Loss of sample due to inadequate and/or improper sealing of the sample container (i.e., leakage of liquids, loss of particulate material from filters, etc.).
- Possible contamination because of inadequate separation of sample types or bulk sample materials (i.e., charcoal tubes or VOA vials shipped in the same container as bulk liquid organics).

- Adequate containment of volatile organic samples and total organic halide samples in septum vials; there should be zero headspace. If headspace is found, this shall be documented. A widely accepted standard for document the approximate size is to judge the headspace to be smaller of larger than a green pea. This should be noted the Checklist or noted in the sample Remarks section of Form DC-1 and the Project Manager/Client Services Group contacted, as the client may wish to re-sample.
- Proper use of special shipping procedures required to preserve the samples. For example, if shipping instructions note that samples are to be kept frozen, then samples should be frozen upon receipt.

If samples are broken or evidence that sample integrity may have been compromised, the incident shall be documented in the designated space of the Checklist and detailed in the Comments section. For CLP samples this shall be documented on Form DC-1, Line 8 and explained in the remarks section for the affected samples and shall also be noted on the Sample Traffic Report Form. The Project Management Group shall be notified of any such problems as soon as possible.

6.0 CONTRACT LABORATORY PROGRAM SAMPLE VESSEL INSPECTION

6.1 For samples submitted by SMO, a review of sample documentation is required once the shipping container has been unpacked. Each shipping container should possess a Chain-of-Custody Form and a Sample Traffic Report Form for the samples contained within the shipping vessel. Each sample should have a Sample Label and a Sample Tag attached to the bottle (where applicable). EPA sample bottles should also have a custody seal over the cap to detect tampering. All items shall be reviewed and documented on Form DC-1.

6.2 Presence or absence of custody seals should be determined. If present, condition of custody seals must be assessed. This shall be documented on Line 1 in the Remarks section of Form DC-1.

6.3 Presence of EPA Sample Tags on sample bottles should be determined. The presence or absence of tags shall be documented on Line 7.

6.4 It shall be determined if the EPA Traffic Report or SAS Packing list was included in the shipment. Documentation shall be made on Line 4.

6.5 It shall be determined if the Chain-of-Custody document was included in the shipment. Documentation shall be made on Line 3.

6.6 The Chain-of-Custody document shall be reviewed to verify the entry of Sample Tag numbers on the Chain-of-Custody. If tag numbers have not been recorded on the Chain-of-Custody, the Sample Coordinator shall perform the document entries and shall document the information in the remarks section for the affected sample.

6.7 The Chain-of-Custody shall be compared with the Sample Labels. Any discrepancies shall be documented. This shall be documented in the remarks section for the affected sample.

6.8 The Chain-of-Custody shall be compared with the Sample Tags. Any discrepancies shall be documented. This shall be documented in the remarks section for the affected sample.

6.9 The Chain-of-Custody shall be compared with the EPA Traffic Reports. Discrepancies shall be documented in the remarks section for the affected sample.

7.0 SAMPLE PRESERVATION INSPECTION

7.1 EPA Contract Laboratory Program (CLP) Samples

7.1.1 Samples which are submitted under the Contract Laboratory Program for inorganic parameters (metals and cyanide) are preserved in the field and may require pH adjustment. These samples are to be checked for proper preservation upon receipt. Because of the nature of these samples proper safety precautions must be observed. Safety glasses, lab coats and disposable gloves shall be worn when handling the sample bottles while performing pH measurements. Gloves shall be changed when soiled to prevent contamination between both the samples or of the employee.

7.1.2 The pH check shall be performed by removing an aliquot of the sample, pouring it into a small disposable cup and depositing the drop onto a pH strip paper. Match the resulting color of the pH paper to the color scale provided with the pH paper. Various ranges of pH papers are used for preservation check. For low range pH measurement, the pH indicator of 0-6 or 0-2.5 will be used. Other wise, pH indicator range 11-13 or 7.5-14 will be used. This will enable to obtain a definitive measurement of pH.

Samples for metals analysis must have a pH of less than 2, and samples for cyanide must have a pH greater than 12.

7.1.3 Record the pH reading on a Sample Preservation Check Documentation Form (Figure 3). The pH may be recorded as less than the value required for acid preserved samples (i.e. <2), or greater than the required value for NaOH preserved samples (i.e. >12 for NaOH preserved cyanide samples). If a sample is discovered to be inadequately preserved, the exact pH reading from the pH check must be recorded. The form shall be signed and dated by the responsible person.

7.1.4 If the pH check indicates that the sample was not adequately preserved, contact the Project Manager immediately to receive instructions from the Sample Management Office. Documentation of sample preservation deficiencies, and instructions given by the Sample Management Office shall be documented in the case file and the case narrative by the Project

Manager. If the Sample Management Office representative dictates that the samples shall be preserved in the laboratory, documentation of this activity shall be indicated on the Sample Preservation Check Documentation Form (Figure 3). The samples may be preserved by Sample Control or laboratory personnel, where applicable. In either case, suitable quantities of the appropriate preservative shall be added to the inadequately preserved samples. Documentation of these activities shall be indicated on the Sample Preservation Check Documentation Form (Figure 3). Copy of the documentation form shall be filed in the case file.

7.1.5 Table 1 provides information on required containers, holding instructions, and holding times for USEPA CLP submitted samples.

7.2 Commercial Samples

7.2.1 EPA requires chemical preservation of water samples to be analyzed for selected parameters. Table 2 presents information which lists parameters, required containers, sample size needed, preservation techniques and holding times for sample analysis performed by methods specified in 40 CFR Part 136, Test Methods for Evaluating Solid Waste - SW 846, and Methods for Chemical Analysis of Water and Wastes (MCAWW). The Sample Coordinator is responsible for verifying that any samples requiring pH adjustment have been appropriately preserved in the field.

7.2.2 The pH check shall be performed by removing an aliquot of the sample, pouring it into a small disposable cup and depositing the drop onto a pH strip paper. Match the resulting color of the pH paper to the color scale provided with the pH paper. Various ranges of pH papers are used for preservation check. For low range pH measurement, the pH indicator of 0-6 or 0-2.5 will be used. Other wise, pH indicator range 11-13 or 7.5-14 will be used. This will enable to obtain a definitive measurement of pH.

Samples for metals analysis must have a pH of less than 2, and samples for cyanide must have a pH greater than 12.

7.2.3 Record the pH reading on a Sample Preservation Check Documentation Form (Figure 3). The pH may be recorded as less than the value required for acid preserved samples (i.e. <2), or greater than the required value for NaOH preserved samples (i.e. >12 for NaOH preserved cyanide samples). As an alternative a check mark may be used to signify that the sample met the preservation criteria. If a sample is discovered to be inadequately preserved the exact pH reading from the pH check must be recorded. The form shall be signed and dated by the responsible person.

7.2.4 If the pH check indicates that the sample was not adequately preserved, contact the Sample Control Supervisor immediately. The Sample Control Supervisor will contact the appropriate Project Manager who will, after consultation with the client, instruct the Sample Control Supervisor of the

appropriate action to follow. Generally, the samples are preserved during that time and the necessary documentation of the problem and the resolution are performed. Also, notations are indicated in the final report describing the situation. Samples may be preserved by Sample Control or laboratory personnel. In either case, suitable quantities of the appropriate preservative shall be added to the inadequately preserved samples. See Table 2 for information on preservatives and criteria. Documentation of these activities shall be made on the Sample Preservation Check Documentation Form (Figure 3). Copy of the documentation form shall be filed in the case file.

Note: If drinking water metals samples are acidified in the laboratory, the time that the samples are acidified must be recorded on the preservation check form (Fig.3) in order to meet the 16 hour hold time requirement.

Note: If Radiochemistry samples are acidified in the laboratory, the time that the samples are acidified must be recorded on the preservation check form (Fig.3) in order to meet the 16 hour hold time requirement.

Note: Samples for Radiochemistry Tritium analysis should not be preserved.

7.2.5 Failure to appropriately preserve the samples in the field may result in invalid analytical data. It is the responsibility of Project Management Group to advise the client of this potential.

All acid preservatives are verified upon arrival to the lab.

7.2.6 Samples for State of North Carolina, which are chlorine sensitive, i.e., organics, ammonia, TKN and cyanide must be checked for the presence of chlorine and treated, if required, before storage and analysis. Documentation of these activities shall be noted on the sample residual chlorine check form (Figure 4).

7.3 pH Adjustment Procedure

7.3.1 Samples which require pH adjustment must have appropriate amounts of the required preservative added. This procedure must be carefully performed and must be properly documented (Figure 3).

7.3.2 Generally, the preservative solutions are relatively concentrated (i.e. 1:1 Nitric or Sulfuric Acid, 5N NaOH) to avoid significant changes in volume. Therefore, only a small quantity of preservative should be added. Due to sample compositions, proper safety precautions must be observed. While performing pH measurements, safety glasses, lab coats and disposable

gloves shall be worn. Gloves shall be changed when soiled to prevent sample contamination.

7.3.3 When a particular sample requires additional preservation the sample shall be opened and a small quantity of the preservative added. The amount of preservative added should be in increments of approximately 0.5ml/250ml of sample. Once the preservative is added the bottle shall be securely capped and shaken to distribute the preservative. The bottle shall then be carefully opened, and observed for evidence of escaping pressure before rechecking the pH. The pH check shall be performed by removing an aliquot of the sample, pouring it into a small plastic cup and depositing the drop onto a strip of pH paper. Match the resulting color of the pH paper to the color scale provided with the pH paper. If the pH does not yet meet the method specification, repeat the process until an acceptable value is maintained.

7.3.4 Documentation of the preservation adjustment and reagent lot number shall be indicated on the Sample Preservation Adjustment Documentation Form (Figure 3). Each space shall be completed for each listed sample and the form shall be signed and dated by the responsible person.

Figure 1

SAMPLE RECEIPT CHECKLIST

W.O. No: _____ Carrier Name: _____

Client Name: _____ Prepared (Logged In) By: _____ / _____
Initials Date

Date Received: _____ Project: _____

Time Received: _____ Site: _____

Received By: _____ VOA Holding Blank I.D. No: _____

	YES	NO		YES	NO
Airbill/Manifest Present?	___	___	Trip Blanks: No. of Sets _____	___	___
			Field Blanks: No. of Sets _____	___	___
No. _____			Equip. Blank: No. of Sets _____	___	___
			Field Duplicate: No. of Sets _____	___	___
Shipping Container in Good Condition?	___	___	MS/MSD: No of Sets_____	___	___
Custody Seals Present on Shipping Container?	___	___	VOA Vials Have Zero Headspace?	___	___
Condition: Broken _____			If no, smaller or greater than a Green Pea (see comments)		
Intact-not dated or signed _____					
Intact-dated and signed _____			Preservatives Added to Sample?	___	___
			pH Check Required?	___	___
Usage of Tamper Evident Type	___	___	Performed By? _____	___	___
Chain-of-Custody Present?	___	___	Ice Present in Shipping Container?	___	___
Chain-of-Custody Agrees with Sample Labels?	___	___	Container # Temp. Container # Temp.		
Chain-of-Custody Signed?	___	___	_____ _____ _____ _____		
Packing Present in Shipping Container?	___	___	_____ _____ _____ _____		
Type of Packing _____			_____ _____ _____ _____		
Custody seals on Sample Bottles?	___	___			
Condition: Good _____ Broken _____			_____ _____ _____ _____		
Total Number of Sample Bottles _____			_____ _____ _____ _____		
Total Number of Samples _____			_____ _____ _____ _____		
Samples Intact?	___	___	_____ _____ _____ _____		
Sufficient Sample Volume for Indicated Test?	___	___	Project Manager Contacted?		
			Name: _____		
			Date Contacted: _____		

Any NO response must be detailed in the comments section below. If items are not applicable to particular samples or contracts, they should be marked N/A/

COMMENTS: _____

_____

_____

_____

Checklist Completed By: _____

Date: _____

Figure 2

SAMPLE LOG-IN SHEET

Lab Name					Page __ of __
Received By (Print Name)					Log-in Date
Received By (Signature)					
Case Number		Sample Delivery Group No.			NRAS Number
Remarks:		EPA Sample #	Aqueous Sample pH	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1. Custody Seal(s)	Present/Absent* Intact/Broken					
2. Custody Seal Nos.	_____ _____					
3. Traffic Reports/Chain of Custody Records or Packing Lists	Present/Absent*					
4. Airbill	Airbill/Sticker Present/Absent*					
5. Airbill No.	_____ _____					
6. Sample Tags	Present/Absent*					
Sample Tag Numbers	Listed/Not Listed on Traffic Report/Chain of Custody Record					
7. Sample Condition	Intact/Broken*/ Leaking					
8. Cooler Temperature Indicator Bottle	Present/Absent*					
9. Cooler Temperature	_____					
10. Does information on Traffic Reports/Chain of Custody Records and sample tags agree?	Yes/No*					
11. Date Received at Lab	_____					
12. Time Received	_____					
Sample Transfer						
Fraction	Fraction					
Area #	Area #					
By	By					
On	On					

* Contact SMO and attach record of resolution

Reviewed By		Logbook No.
Date		Logbook Page No.

Figure 3

Sample Preservation Check Documentation Form

Work Order: ____________________

Parameter:	Metals (*A)	Phenol	TPH O&G	Classical Parameters	Cyanide	Sulfide	Radiology (*B)	Other Preservation Adjustment	Reagent Lot #	see checklist for comments
Preservative:	HN03	H2SO4	H2SO4	H2SO4	NaOH	NaOH	HN03			
pH Value	<2	<2	<2	<2	>12	>9	<2			
GPL ID										
								Y / N		
								Y / N		
								Y / N		
								Y / N		
								Y / N		
								Y / N		
								Y / N		
								Y / N		
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								Y / N		
								Y / N		
								Y / N		
								Y / N		
								Y / N		
								Y / N		
								Y / N		

(*A). If drinking water metals sample is acidified in the laboratory,
the TIME that the samples are acidified MUST be recorded in order to meet the 16 hour hold time requirement.
(*B). If Radiochemistry sample is acidified in the laboratory,
the TIME that the samples are acidified MUST be recorded in order to meet the 16 hour hold time requirement.
Tritium analysis should NOT be preserved.

Sample Preservation Check Performed By: ____________________ Date: ____________________

Figure 4

North Carolina Sample Residual Chlorine Check Form

Work Order: _____

Client ID	Organics	TKN	Ammonia	Cyanide	Comments

Sample Preservation Check Performed By: _____ Date: _____

Table 1
Summary of Contract Laboratory Program Required
Containers, Preservation Techniques, and Holding Times[1]

Parameter	Container	Holding Instructions	Maximum Sample Holding Time
Metals	P, G	Room Temperature	180 days from VTSR
Mercury	P, G	Room Temperature	26 days from VTSR
Cyanide (Total and amenable to chlorination)	P, G	Room Temperature	12 days from VTSR
Volatiles	G(TS)	Store at 4° ± 2°C	10 days from VTSR
Semivolatiles (water)	G(TC)	Store at 4° ± 2°C (in dark)	5 days from VTSR [a]
Semivolatiles (soil/sediment)	G(TC)	Store at 4° ± 2°C (in dark)	10 days from VTSR [a]
Pesticides/PCBs (water)	G(TC)	Store at 4° ± 2°C (in dark)	5 days from VTSR [a]
Pesticides/PCBs (soil/sediment)	G(TC)	Store at 4° ± 2°C (in dark)	10 days from VTSR [a]

[a] - Extract holding time is 40 days after extraction
P = plastic
G = glass
G(TC) = glass, TFE-lined cap
G(TS) = glass, TFE-lined septum

[1] - USEPA Contract Laboratory Program; Statement of Work for Inorganics Analysis; Multi-media, Multi-concentration; SOW No. 3/90

USEPA Contract Laboratory Program; Statement of Work for Organics Analysis; Multi-media, Multi-concentration; SOW No. 3/90

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container[1]	Minimum Sample Size (ml)	Preservation[2,3]	Maximum Holding Time[4]
BACTERIAL TESTS				
Coliform, fecal and total	P, G (sterile)	200	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	6 hours
Fecal Streptococci	P, G (sterile)	200	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	6 hours
INORGANIC TESTS[7]				
Acidity	P, G	200	Cool, 4°C	14 days
Alkalinity	P, G	200	Cool, 4°C	14 days
Ammonia	P, G	200	Cool, 4°C, H_2SO_4 to pH < 2	28 days
Biochemical Oxygen Demand	P, G	1,000	Cool, 4°C	48 hours
Bromide	P, G	250	None	28 days
Chloride	P, G	50	None Required	28 days
Chlorine, total residual	P, G	500	None Required	Analyze immediately
Color	P, G	500	Cool, 4°C	48 hours

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container[1]	Minimum Sample Size (ml)	Preservation[2,3]	Maximum Holding Time[4]
Cyanide, total and amenable to chlorination	P, G	1,000	Cool, 4°C, H_2SO_4 to pH > 12, 0.6g ascorbic acid[5]	14 days[6]
Fluoride	P	1,000	None Required	28 days
Hardness	P, G	200	HNO_3 or H_2SO_4 to pH < 2	6 months
Hydrogen ion (pH)	P, G	250	None Required	Analyze Immediately
3jeldahl and organic nitrogen	P, G	500	Cool, 4°C, H_2SO_4 to pH < 2	28 days
Chromium VI	P(A), G(A)	300	Cool, 4°C	24 hours
Mercury	P(A), G(A)	500	HNO_3 to pH < 2	28 days
Metals, except Chromium VI and Mercury	P(A), G(A)	500	HNO_3 to pH < 2	6 months
Nitrate	P, G	200	Cool, 4°C	48 hours
Nitrate-nitrite	P, G	200	Cool, 4°C, H_2SO_4 to pH < 2	28 days
Nitrite	P, G	200	Cool, 4°C	48 hours

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container[1]	Minimum Sample Size (ml)	Preservation[2,3]	Maximum Holding Time[4]
Oil and Grease	G, (wide mouth)	2000	Cool, 4°C, H_2SO_4 to pH < 2	28 days
Organic Carbon	P, G	100	Cool, 4°C, H_2SO_4 to pH < 2	28 days
Orthophosphate	P(A), G(A)	200	Filter Immediately, Cool 4°C	48 hours
Oxygen, dissolved (probe)	G Bottle and Top	300	None Required	Analyze Immediately
Winkler	G Bottle and Top	300	Fix on site and store in dark	8 hours
Phenols	G only	1000	Cool, 4°C, H_2SO_4 to pH < 2	28 days
Phosphorous, elemental	G	200	Cool, 4°C	48 hours
Phosphorous, total	P, G	200	Cool, 4°C, H_2SO_4 to pH < 2	28 days
Residue, total	P, G	200	Cool, 4°C	7 days
Residue, filterable (TDS)	P, G	200	Cool, 4°C	48 hours
Residue, nonfilterable (TSS)	P, G	200	Cool, 4°C	7 days
Residue, settleable	P, G	200	Cool, 4°C	48 hours

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container[1]	Minimum Sample Size (ml)	Preservation[2,3]	Maximum Holding Time[4]
Residue, volatile	P, G	200	Cool, 4°C	7 days
Silica	P	500	Cool, 4°C	28 days
Specific Conductance	P, G	500	Cool, 4°C	28 days
Sulfate	P, G	50	Cool, 4°C	28 days
Sulfide	P, G	200	Cool, 4°C, add zinc acetate plus sodium hydroxide to pH > 9	7 days
Sulfite	P, G	100	None Required	Analyze Immediately
Surfactants	P, G	500	Cool, 4°C	48 hours
Temperature	P, G	250	None Required	Analyze
Turbidity	P, G	250	Cool, 4°C	48 hours

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container[1]	Minimum Sample Size (ml)	Preservation[2,3]	Maximum Holding Time[4]
ORGANIC TESTS[8]				
Purgeable Halocarbons	G, Tef.-lined septum	2X 40	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable aromatic hydrocarbons	G, Tef.-lined septum	2X 40	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], HCl to pH 2[9]	14 days
Acrolein and acrylonitrile	G, Tef.-lined septum	2X 40	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], Adjust pH to 4-5[10]	14 days
Phenols[11]	G, Tef.-lined cap	2000	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11]	G, Tef.-lined cap	2000	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Tef.-lined cap	2000	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[12,14]	G, Tef.-lined cap	2000	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11] acrylonitrile	G, Tef.-lined cap	2500	Cool, 4°C	7 days until extraction; 40 days after extraction

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container[1]	Minimum Sample Size (ml)	Preservation[2,3]	Maximum Holding Time[4]
Nitroaromatics and isophorone[11]	G, Tef.-lined cap	2000	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]. Store in dark	7 days until extraction; 40 days after extraction
Polynuclear aromatic hydrocarbons[11]	G, Tef.-lined cap	2000	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction 40 days after extraction
Haloethers[11]	G, Tef.-lined cap	2000	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Chlorinated hydrocarbons[11]	G, Tef.-lined septum	2X 40	Cool, 4°C	7 days until extraction; 40 days after extraction
PESTICIDES				
Pesticides[11]	G, Tef.-lined cap	1000	Cool, 4°C, pH 5-9[15]	7 days until extraction 40 days until extraction
RADIOLOGICAL TESTS				
Alpha, beta, radium	P, G	2000	HNO_3 to pH 2	6 months

1. Polyethylene (P) or Glass (G), G(A) or P(A) = rinsed with 1:1 Nitric Acid

2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

3. When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight 0000000or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator under 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See 136.3(e) for details.

5. Should only be used in the presence of residual chlorine.

6. Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

7. Samples should be filtered immediately on-site before adding preservative for dissolved metals.

8. Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

9. Sample receiving no pH adjustment must be analyzed within 7 days of sampling.

10. The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

11. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forth days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine), and footnotes 12 and 13 (re: the analysis of benzidine).

12. If 1,2-Diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

13. Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

14. For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

15. The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

SOP No: Q.6

Title: SOP for Method 8081A
Organochlorine Pesticides

1.0 SCOPE AND APPLICATION

1.1 Method 8081A is used to determine the concentration of various organochlorine pesticides in extracts from solid and liquid matrices. Table 1 indicates compounds that may be determined by this method.

TABLE 1

Target Compound	
Alpha-BHC	Diallate
Gamma-BHC (Lindane)	Isodrin
Heptachlor	Kepone
Aldrin	Toxaphene
Beta-BHC	Tetrachloro-m-xylene (Sur)
Delta-BHC	Decachlorobiphenyl
Heptachlor Epoxide	
Endosulfan I	
4,4'-DDE	
Dieldrin	
Endrin	
Endosulfan II	
4,4'-DDD	
4,4'-DDT	
Endrin Aldehyde	
Endosulfan Sulfate	
Methoxychlor	
Endrin Ketone	
Alpha-Chlordane	
Gamma-Chloridane	
Hexachlorobenzene	
Hexachlorocyclopentadiene	
Chlorobenryilate	

1.2 The following compounds may also be determined using this method:

Compound Name	CAS Registry No.
Arochlor	15972-60-8
Captafol	2425-06-1
Captan	133-06-2
Chloroneb	2675-77-6
Chloropropylate	99516-95-7
Chlorothalonil	1897-45-6
DCPA	1861-32-1
Dichlone	117-80-6
Dicofol	115-32-2
Etridiazole	2593-15-9
Halowax-1000	58718-66-4
Halowax-1001	58718-67-5
Halowax-1013	12616-35-2
Halowax-1014	12616-36-3
Halowax-1051	2234-13-1
Halowax-1099	39450-05-0
Mirex	2385-85-5
Nitrofen	1836-75-5
PCNB	82-68-8
Perthane	72-56-0
Propachlor	1918-16-17
Strobane	8001-50-1
Trans-Nonaroclor	39765-80-5
Trans-Permethrn	51877-74-8
Trifluralin	1582-09-8

2.0 SUMMARY OF METHOD

2.1 Method 8081A provides gas chromatographic conditions for the detection of ppb levels of certain organochlorine pesticides. Prior to the use of this method, appropriate sample extraction techniques must be used.

3.0 INTERFERENCES

3.1 Interferences by phthalate esters can pose a major problem in pesticide determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large-eluting peaks, especially in the 15% and 50% fraction from cleanups. Avoiding contact with any plastic materials can best minimize interferences from pthalates.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph

Hewlett Packard GC Systems 5890 equipped with autosampler is used. EnviroQuant software systems for data recording and processing is interfaced with the GC system.

4.2 Columns

4.2.1 Dual column connected with a "Y" connector to a single injection port is used. In this mode, material injected is split between the columns and detected by 2 separate ECD detectors.

4.2.2 Column 1: 30 meter, 5% opphenyl DI Methyl Polysiloxane (RTX-CLP) fused silica column is used.

4.2.3 Column 2: 30 meter, 14% cyanopropyl methyl polysiloxane (RTX-CLP2) fused silica column is used.

4.2.4 5 meter guard column (inert) is used at the "Y" connector.

4.3 GC Condition

4.3.1 The following GC parameters are implemented when Pest/PCBs are analyzed:

- Injection port temperature 245°C
- Detectors temperature 300°C
- A tri-ramp temperature program is used

1st Ramp:	
Initial temperature	150°C
Initial time	0.5 min
Rate	8°C/min
Final temperature	180°C
Final time	10 min

2nd Ramp:	
Initial temperature	180°C
Rate	8°C/min
Final temperature	210°C
Final time	10 min

3rd Ramp:	
Initial temperature	210°C
Rate	15°/min
Final temperature	270°C
Final time	8 min

- Equilibrium time 0.75 min
- Septum purge on at 0.75 min at about 2ml/min
- Attenuation 0
- Signal rages 0
- Splitless purge 50ml/min
- Makeup gas 80ml/min

4.4 Gases

4.4.1 Helium ultrapure grade is used as a gas carrier at about 5ml per min at 50° measured at the column end.

4.4.2 Make up gas is Argon/Methane (5% Methane) and the flow rate is about 80 ±5ml per min for each detector.

5.0 CALIBRATION

5.1 Calibration standards are prepared at five concentration levels through dilution of the stock standards with hexane (refer to standard logbook). Concentrations of the five level calibration are listed on Table 3.

TABLE 3

Compound	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)
4,4'-DDD	5	25	50	75	100
4,4'-DDE	5	25	50	75	100
4,4'-DDT	5	25	50	75	100
Aldrin	5	25	50	75	100
alpha-BHC	5	25	50	75	100
beta-BHC	5	25	50	75	100
Alpha-Chlordane	5	25	50	75	100
Gamma-Chlordane	5	25	50	75	100
Decachlorobiphenyl (CB Surr)	2.5	12.5	25	37.5	50
delta-BHC	5	25	50	75	100
Tetrachloro-m-xylene (Surr)	2.5	12.5	25	37.5	50
Dieldrin	5	25	50	75	100
Endosulfan I	5	25	50	75	100
Endosulfan II	5	25	50	75	100
Endosulfan Sulfate	5	25	50	75	100
Endrin	5	25	50	75	100
Endrin aldehyde	5	25	50	75	100
Endrin ketone	5	25	50	75	100
Gamma-BHC (Lindane)	5	25	50	75	100
Heptachlor	5	25	50	75	100
Heptachlor epoxide	5	25	50	75	100
Methoxychlor	5	25	50	75	100
Toxaphene			500		

5.2 Surrogate standards Tetrachloro-m-xylene and Decachlorobiphenyl are used. They are calibrated at levels indicated on Table 3.

6.0 GC ANALYSIS

6.1 Retention time windows

6.1.1 Make 3 injections of midpoint standard mixtures and multipeaks throughout the course of a 72-hour period. Calculate standard deviation of the three absolute retention times for each single component standard. For multiresponse products, choose one major peak from the cluster and calculate the standard deviation of the three retention times for that peak.

6.1.1.1 Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define retention time window. For multipeak products, primarily combination of chromatography pattern and retention times are used.

6.1.1.2 In those cases where the standard deviation for a particular standard is zero, substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

6.1.1.3 When a new GC column is installed, retention time window must be established.

6.2 Degradation of DDT and Endrin

6.2.1 Check for degradation problems by injecting a EVAL MIX containing 4,4'-DDT and endrin. Look for the degradation products of 4,4'-DDT (4,4'-DDE and 4,4'-DDD) and endrin (endrin ketone and endrin aldehyde). Degradation must be less than 15% before sample analysis.

$$\%\ \text{Breakdown for DDT} = \frac{\text{Total DDT degradation peak area (DDE + DDD) x 100}}{\text{Total DDT peak area (DDT + DDE + DDD)}}$$

$$\%\ \text{Breakdown for Endrin} = \frac{\text{Total Endrin degradation peak area (endrin aldehyde+endrin ketone) x 100}}{\text{Total Endrin peak area (endrin+endrin ketone+endrin aldehyde)}}$$

6.2.2 At the beginning of each day, break down of Endrin and DDT must be in control before sample analyses should commence. When compounds don't meet the required criteria, as a corrective action for the breakdown, clipping portion of the column and injection port clean up will be performed. Also, to reduce some of the contaminations built up, the gold seal will be replaced.

6.3 Calibration

6.3.1 Five point calibration of pesticides and single point calibration for toxaphene and chlordane are initially analyzed. After analyzing the initial calibration standards, average response factor (CF) should be calculated for each compound. The percent relative standard deviation %RSD should be equal or less than 20% for each compound. It is likely that some analytes may exceed the 20% acceptance limit for the %RSD. In those instances the following steps should be considered:

- If the %RSD is greater than 20%, the analyst should review the results (area counts, calibration factors, standard concentrations) for those analytes to ensure that the problem is not associated with a single standard. If so, that standard should be reanalyzed and RSD recalculated. Replacing of standard may be necessary in some cases.

- It may also be necessary to narrow the calibration ranges to achieve a better linearity. High standard may saturate the column or/and the detector and need to be dropped from the calibration curve accordingly. Similarly poor purging compounds that exhibited erratic chromatographic behavior in the lowest calibration point could also be reviewed and dropped if necessary. The changes to the upper end of the calibration will affect the need to dilute samples above that range, while the change to the lower calibration will affect the sensitivity and elevate the reporting limit of the method. When considering dropping one or two points to narrow the calibration range, a minimum of five points is required for curve to be acceptable.

After visual inspection of the calibration points for each analytes and for those analytes that did not meet the %RSD criteria, at the discretion of the analyst it should be considered to employ a regression equation that does not through the origin or a quadratic (second order – requires six standards) model. Linearity is presumed acceptable if the correlation coefficient (r) is equal to or greater than 0.995 or the coefficient of determination (r^2) is equal to or greater than 0.99.

6.3.2 Daily continuing calibration: degradation check, mid-level pesticide, 8081 other compounds, chlordane and toxaphene are analyzed at the beginning of each shift. Percent difference from the mean CF calculated from the ICAL should be within ±15% for all compounds except for surrogate.

6.3.3 Sequence continuing standard: mid-level pesticide should be injected between every ten injections of samples and/or QC and at the end of every 12 hours, whichever is more frequent. All samples that were injected after the standard exceeding the criteria of linearity must be re-injected if the initial analyses indicate the presence of specific target analyte that exceeded the criteria. However, if the standard analyzed after a group of samples exhibited a response for an analyte above 15%

limit, and if the analyte was not detected in the specific samples, re-analysis is not necessary. In contrast, if the response of the instrument exhibited 15% below the initial calibration response, then re-injection of sample is necessary (whether an analyte was detected or not-detected). Experience of the chromatographer is an influential factor in the determination of sample re-analysis.

6.3.4 A mid-level calibration verification standard prepared from a different source is injected following the five-point calibration. Recovery range should be within ±20%. If acceptance criteria was not met, investigate the problem. Re-injection of a new 5-point initial calibration may be necessary.

6.3.5 When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of manual integration and notation of date and initials of person performing the task.

6.4 Sample Collection, Preservation and Handling

6.4.1 Sample extracts are preserved by keeping them cooled to 4°C

6.4.2 The holding times for extraction of the samples are 7 days of sampling for water samples, and 14 days of sampling for soil samples. If sample has been extracted outside the holding time, not the aberration on the sample non-conformity report, and notify the manager for further instruction.

6.4.3 Sample must be analyzed within 40 days of the extraction. If sample has been analyzed outside the holding time, note the aberration on the sample non-conformity report, and notify the manager.

6.5 Sample Analysis

6.5.1 Prime the GC column with 1ppm pesticides standard if the instrument has not been used for more than 12 hours followed by an instrument blank.

6.5.2 When all degradation and calibration requirements are met sample analysis may begin. After each batch of 10 runs, linearity should be checked before any more samples are analyzed. Refer to 7.0 for quality control requirements.

6.5.3 Dilution must be made if the response of any compound exceed the highest calibration standard in the curve.

6.5.4 Peak identification is primarily based on detection on both columns within the established retention time. When results are confirmed using two dissimilar columns, the agreement between the quantitative results should be evaluated after identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula below.

$$RPD = \frac{|R1 - R2|}{(R1+R2)/2} \times 100$$

where R1 and R2 are the results on the two columns and the vertical bars in the equation above indicate the absolute value of the difference. Therefore, the RPD is always a positive value.

6.5.4.1 If one result is significantly higher (e.g., > 40%), check the chromatogram for anomalies. If there is no evidence of chromatographic problem, report the higher result and noted in the case narrative. The experience of the analyst may prove invaluable in determining whether the results are confirmed or not based on retention times and agreement between quantitation results of both columns.

6.5.4.2 When results are confirmed using a second GC column, the higher of the two column results should be reported, unless the column associated with the higher results did not pass the acceptance criteria. In such cases, the lower value that is associated with the column that did pass the criteria may be reported. The experience of the analyst may prove invaluable in determining which results from which column will be reported.

6.5.5 GCMS confirmation may be implemented if the concentration permits.

6.6 Cleanup

6.6.1 If peak detection and identification of pesticides are prevented due to interferences of steroids, esters, ketones, glycerides and some hydrocarbons, the extract may need to undergo florisil cleanup using Method 3620B. If peak detection of pesticides are prevented by sulfur interferences which appear as huge hump at the beginning of the chromotogram, the extract may need to undergo sulfur cleanup (Method 3660B).

6.7 Low Concentration Method

6.7.1 This method is applicable to soil and water samples containing low level contaminants with minimal matrix interferences. To achieve low detection limits, the final volumes of sample extracts are brought to 2.0ml. Concentrations of surrogate and matrix spike standards should be adjusted before extraction in order to produce levels comparable to low level procedure. Quality control and recovery ranges are not expected to be affected by final volume reduction and low level quality control criteria should be applicable to this modified method.

6.8 Calculation

6.8.1 All quantifications are based on external standard calculations.

6.8.1.1 Calculation for calibration factor

$$\text{Calibration factor} = \frac{\text{Total Area of Peak*}}{\text{Mass injected (nanograms)}}$$

* for multipeak analytes use total areas of all designed peaks for quantitation.

6.8.1.2 Percent difference

$$\text{Percent difference} = \frac{(R_1 - R_2) \times 100}{R_1}$$

R_1 = Calibration factor from first analysis
R_2 = Calibration factor from succeeding analyses

6.8.1.3 The concentration of each analyte in the sample may be determined by calculating the amount of standard injected from the peak response, using the calibration curve or factor determined from 6.7.1.1.

Aqueous Concentration (ug/L) = $[(A_x)(A)(Vt)(D)]/[(As)(Vi)(Vs)]$

Solid Concentration (ug/kg) = $[(A_x)(D)(A)(Vt)]/[(As)(Ws)(Vi)]$

Where:

A_x = Response for the analyte in the sample, (area or peak height
A = Amount of standard injected in ng
As = Response for the external standard
Vi = Volume of extract injected
D = Dilution factor, if any
Vt = Volume of total extract
Vs = Volume of sample extracted
Ws = Weight of sample extracted

For non-aqueous samples, the unit is µg/kg and dry weight of sample is used for Ws.

6.8.1.4 For DOD projects, when results are confirmed using a second column, the calibration criteria for both columns are the same and must be met in order for the analysis to be valid.

7.0 QUALITY CONTROL

7.1 Required Instrument QC

7.1.1 It is required that the % RSD vary by ≤20% when 5 point calibration factors are compared.

7.1.2 It is required that difference of daily response of a given analyte vary by ± 15% when compared to initial responses. If criteria were not met either on the basis of each compound or the average across all compounds, check instrument conditions, re-inject another mid-level calibration standard, and if necessary, analyze a new 5-point calibration.

7.1.3 All succeeding standards in an analysis sequence must fall within the daily retention time window established by the first standard of the sequence. If retention time shifted, perform regular maintenance on the instrument and re-inject the mid-level standard. If the system is still unstable, the problem should be corrected before any further samples can be analyzed.

7.1.4 Control limits for MS/MSD, LCS and surrogates are established semi-annually for internal use.

7.1.5 For DOD projects, recoveries of LCS, MS/MSD and surrogates are evaluated against DOD QSM control limits policy.

7.2 Method Detection Limits

7.2.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

7.3 Method Performance

7.3.1 The MDL concentrations listed in the GPL MDL table are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

7.4 Matrix Spikes

7.4.1 MS and MSD must be analyzed with each batch of up to 20 samples of the same matrix processed together. If less than twenty samples are analyzed per month, MS/MSD must be analyzed on per month basis. Percent recoveries and Relative Percent Difference (RPD) should be calculated as follows:

$$\text{Matrix Spike \% Recovery} = \frac{SSR-SR}{SA} \times 100$$

Where:

SSR = Spike Sample Results

SR = Sample Results

SA = Spike Added from Spiking Mix

$$RPD = \frac{D1 - D2}{(D1 + D2)/2} \times 100$$

D1 = First Sample Value

D2 = Second Sample Value

The MS/MSD is evaluated by comparing the precision of target analytes to the recovery windows established. MS/MSD data evaluation is more complex than method blank or LCS data since MS/MSD measure matrix effect in addition to sample preparation and analysis error. MS/MSD that fail to meet the acceptance criteria would indicate that a potential matrix effect is present. The laboratory must assess the batch to determine whether the spike results are attributable to matrix affect, or the result of another problem in the analytical process. If all the QC batch elements, which are not affected by the sample matrix, are in control (e.g., method blank, LCS), and if there is no evidence that the spiking was not properly performed, the poor spike recovery may be attributed to matrix effect. If the LCS compounds that are not affected by the sample matrix are out of control, and if the same compounds in the MS/MSD are outside control limit, then matrix spiked sample(s) must be re-processed through the entire analytical procedure.

7.5 Lab Control Sample (LCS)

7.5.1 A control check sample is extracted and analyzed at frequency as the method blank with each extracted batch. LCS compound must be within the established control limits. Statistical control limit are based on minimum of 20 data points. Data points used in the data set must not be selectively included or excluded. If recovery of LCS compound falls outside the established limit, corrective action must be taken. After corrective action, if LCS analyte recovery is still outside QC acceptance limits, re-extraction of the batch of samples may be necessary if the holding times have not elapsed and/ or refer to supervisor. Should any LCS be out of control, the lab must re-extract and re-analyze for all compounds that had criteria failed. In-house established limits for LCS should be in the range of ±20%. When the results of the matrix spikes indicate a matrix problem, the LCS results are used to verify the laboratory performance in a clean matrix.

7.5.2 Each LCS must be evaluated against the Control Limits and Marginal Exceedance limit. If any LCS is outside the Control Limit, it should be also compared to the Laboratory Marginal Exceedance Limits to ensure that it does not exceed. If a single analyte in the LCS exceeded the Marginal Exceedance Limit, the LCS had failed and corrective action needs to be initiated.

7.5.3 If the LCS has more than the allowable number of Marginal Exceedance, the LCS has failed and corrective action needs to be conceded.

7.5.4 The corrective action for failed LCS is based on professional judgment in conjunction with matrix spike and surrogate recoveries in the same batch. If after checking the associated QC's it was determined by the section supervisor or manager that the LCS had failed, all affected samples associated with the out of control LCS should be reprocessed and re-analyzed

7.5.5 If samples cannot be reprocessed due to lack of sample volume or the holding times has lapsed, the results should be reported with appropriate flag. The report case narrative must include a discussion of the failed LCS, and its impact on the data quality.

7.5.6 When LCS is spiked with large number of analytes, the laboratory should add up total number of exceedances for the LCS based on the number of analyte spiked in the LCS. The total of exceedance should be compare with the allowable number from the following chart.

Number of analyte in the LCS	Allowable Number of Marginal Exceedances of LCS
< 11	0
11-30	1
31-50	2
51-70	3
71-90	4
>90	5

7.5.7 Laboratory Control Spike for toxaphene and chlordane should be analyzed quarterly unless it is requested sooner by a client.

7.6 Blanks

7.6.1 Blank is extracted and analyzed with each analytical batch. Blanks must be contaminant free. Concentration of any confirmed peak should be less than one half of the reporting limit.

7.7 Surrogates

7.7.1 Percent recoveries for the surrogates determined by plotting percent recoveries of surrogates measured in 20 consecutive blanks. Control limits for each surrogate compound is measured using the following formula:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p – 3s
Where p is the mean recovery and s is the standard deviation.

7.7.2 Two surrogates (TCX and DCB) are added to each sample, however, one surrogate can be acceptable for QC determination. Calculate surrogate standard recovery on all samples, and all QC samples. Determine if the recovery is within limits. If recovery is not within limits, re-extract and re-analyze the sample and/or refer to supervisor.

7.7.3 For DOD projects, surrogate recoveries should be evaluated against DOD QSM control limits policy(Appendix DOD-D).

8.0 SAFETY

8.1 Safety glasses for eye protection, laboratory coats for body protection, latex gloves for hand protection.

8.2 Due to the toxicity or carcinogenicity of each reagent, each chemical compound should be treated as a potential health hazard.

8.3 Material safety Data Sheets (MSDS) can be found on the procurement bookshelves located in the company library.

8.4 Preparation of the standard should be handled under a hood.

9.0 POLLUTION PREVENTION

9.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

10.0 WASTE MANAGEMENT

10.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

11.0 DEFINITIONS

11.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

12.0 REFERENCES

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Revision 1, December 1996.

- DOD Quality System Manual, Final Version 3, March 2005

SOP No: Q.7

Title: SOP for Method 8082
PCBs, PCTs and PCB Congeners

1.0 SCOPE AND APPLICATION

1.1 Method 8082 is used to determine the concentrations of various polychlorinated biphenyls (PCBs), polychlorinated terphenyls (PCTs), or as individual PCB congeners in various matrices such as aqueous, solids, oils, products and wipes. Table 1 indicates compounds that may be determined by this method.

TABLE 1

Target Compounds
2-Chlorobiphenyl
2,3-Dichlorobiphenyl
2,2',5-Trichlorobiphenyl
2,4',5-Trichlorobiphenyl
2,2',5,5'-Tetrachlorobiphenyl
2,3',4,4'-Tetrachlorobiphenyl
2,2',3,4,5'-Pentachlorobiphenyl
2,2',4,5,5'-Pentachlorobiphenyl
2,3,3',4',6-Pentachlorobiphenyl
2,2',3,4,4',5'-Hexachlorobiphenyl
2,2',3,4,5,5'-Hexachlorobiphenyl
2,2',3,5,5',6-Hexachlorobiphenyl
2,2',4,4',5,5'-Hexachlorobiphenyl
2,2',3,3',4,4',5-Heptachlorobiphenyl
2,2',3,4,4',5,5'-Heptachlorobiphenyl
2,2',3,4,4',5',6-Heptachlorobiphenyl
2,2',3,4',5,5',6-Heptachlorobiphenyl
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl
2,2',3,3',4,4',5,6-Octachlorobiphrnyl
2,2',3,3',4,4'-Hexachlorobiphenyl
2,2',3,5'-Tetrachlorobiphenyl
2,3,4,4',5-Pentachlorobiphenyl
2,3',4,4',5-Pentachlorobiphenyl
2,3',4,4',5,5'-Hexachlorobiphenyl

2,3,3'4,4',5'-Hexachlorobiphenyl
2,3,3',4,4',5,5'-Heptachlorobiphenyl
2,3,3',4,4',5-Hexachlorobiphenyl
2,3,3',4,4'-Pentachlorobiphenyl
2,4'-Dichlorobiphenyl
2,4,4'-Trichlorobiphenyl
3,3',4,4'-Tetrachlorobiphenyl
3,3',4,4',5-Pentachlorobiphenyl
3,3',4,4'5,5'-Hexachlorobiphenyl
3,4,4',5-Tetrachlorobiphenyl
Aroclor 1016
Aroclor 1260
Aroclor 1221
Aroclor 1232
Aroclor 1242
Aroclor 1248
Aroclor 1254
Aroclor 5432
Aroclor 5460
Aroclor 6040
Aroclor 6062
Aroclor 6070
Tetrachloro-m-xylene(Sur)
Decachlorobiphenyl (Sur)
Hexabromobiphenyl (Sur)

2.0 SUMMARY OF METHOD

2.1 Method 8082 provides gas chromatographic conditions for the detection of ppb levels of certain polychlorinated biphenyls, polychlorinated terphenyls and congeners.

2.2 Prior to the use of this method, appropriate sample extraction techniques must be used.

3.0 INTERFERENCES

3.1 Interferences by phthalate esters can pose a major problem in PCB determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large-eluting peaks, especially in the 15% and 50% fraction from cleanups. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph

Hewlett Packard GC Systems 5890 equipped with autosampler is used. EnviroQuant software systems for data recording and processing is interfaced with the GC system.

4.2 Columns

4.2.1 Dual column connected with a "Y" connector to a single injection port is used. In this mode, material injected is split between the columns and detected by 2 separate ECD detectors.

4.2.2 Column 1: 30 meter, 5% opphenyl DI Methyl Polysiloxane (RTX-CLP) fused silica column is used.

4.2.3 Column 2: 30 meter, 14% cyanopropyl methyl polysiloxane (RTX-CLP2) fused silica column is used.

4.2.4 5 meter guard column (inert) is used at the "Y" connector.

4.3 GC Condition

4.3.1 The following GC parameters are implemented when PCBs are analyzed:

- Injection port temperature 200°C
- Detectors temperature 300°C
- A double ramp temperature program is used

1st Ramp:	
Initial temperature	150°C
Initial time	1.0 min
Rate	15°C/min
Final temperature	200°C
Final time	4.0 min

2nd Ramp:	
Initial temperature	200°C
Rate	10°C/min
Final temperature	300°C
Final time	5.0 min

- Equilibrium time 0.75 min
- Septum purge on at 0.75 min at about 2ml/min
- Attenuation 0
- Signal rages 0
- Splitless purge 50ml/min
- Makeup gas 80ml/min

4.3.2 The following GC parameters are implemented when PCTs are analyzed:

Injector Temp	200°C
Detector Temp	300°C
Initial Temp	150°C
Initial Time	1.0 min.
Ramp Rate	15°/min.
Final Temp	300°C
Final Time	15 min.

The Electronic Pressure Controller should be programmed as follows:

Initial Pressure Time	20min.
Initial Pressure	8 PSI
Rate	99 PSI
Final Pressure	25 PSI
Final Time	13min.

4.4 Gases

4.4.1 Helium ultrapure grade is used as a gas carrier at about 5ml per min at 50° measured at the column end.

4.4.2 Make up gas is Argon/Methane (5% methane) and the flow rate is about 80 ± 5ml per min for each detector.

5.0 CALIBRATION AND STANDARDIZATION

Calibration standards are prepared at five concentration levels through dilution of the stock standards with hexane (refer to standard logbook). Concentrations of the five level calibration and single point Aroclor are listed on Table 2.

5.1 Aroclors

To demonstrate the linearity of the detector, for PCBs a five-point concentration of a mixture of Aroclor 1016 and Aroclor 1260 should be analyzed. Mid-level point of other five Aroclor (listed on Table 2) are required to aid in pattern recognition and single point calibration.
For PCTs, a five point concentration of standards are analyzed
(listed on Table 2) and calibration curves for each target analyte is established.

5.2 PCB Congeners

If samples are to be determined for individual PCB congeners, prepare a minimum of five concentrations of PCB congeners.

TABLE 2

Compound	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)
2-Chlorobiphenyl	2	5	25	50	75	100
2,3-Dichlorobiphenyl	2	5	25	50	75	100
2,2',5-Trichlorobiphenyl	2	5	25	50	75	100
2,4',5-Trichlorobiphenyl	2	5	25	50	75	100
2,2',3,5'-Tetrachlorobiphenyl	2	5	25	50	75	100
2,2',5,5'-Tetrachlorobiphenyl	2	5	25	50	75	100
2,3',4,4'-Tetrachlorobiphenyl	2	5	25	50	75	100
2,2',3,4,5'-Pentachlorobiphenyl	2	5	25	50	75	100
2,2',4,5,5'-Pentachlorobiphenyl	2	5	25	50	75	100
2,3,3',4',6-Pentachlorobiphenyl	2	5	25	50	75	100
2,2',3,4,4',5'-Hexachlorobiphenyl	2	5	25	50	75	100
2,2',3,4,5,5'-Hexachlorobiphenyl	2	5	25	50	75	100
2,2',3,5,5',6-Hexachlorobiphenyl	2	5	25	50	75	100
2,2',4,4',5,5'-Hexachlorobiphenyl	2	5	25	50	75	100
2,2',3,3',4,4',5-Heptachlorobiphenyl	2	5	25	50	75	100
2,2',3, 4,4',5,5'-Heptachlorobiphenyl	2	5	25	50	75	100
2,2',3,4,4',5',6-Heptachlorobiphenyl	2	5	25	50	75	100
2,2',3,4',5,5',6-Heptachlorobiphenyl	2	5	25	50	75	100
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	2	5	25	50	75	100
2,3,4,4',5-Pentachlorobiphenyl	2	5	25	50	75	100
2,2',3,3',4,4',5,6-Octachlorobiphrnyl	2	5	25	50	75	100
2,2',3,3',4,4'-Hexachlorobiphenyl	2	5	25	50	75	100
2,2',3,5'-Tetrachlorobiphenyl	2	5	25	50	75	100
2,3',4,4',5,5'-Hexachlorobiphenyl	2	5	25	50	75	100
2,3',4,4',5-Pentachlorobiphenyl	2	5	25	50	75	100
2,3,3'4,4',5'-Hexachlorobiphenyl	2	5	25	50	75	100
2,3,3',4,4',5,5'-Heptachlorobiphenyl	2	5	25	50	75	100
2,3,3',4,4',5-Hexachlorobiphenyl	2	5	25	50	75	100
2,3,3',4,4'-Pentachlorobiphenyl	2	5	25	50	75	100
2,4'-Dichlorobiphenyl	2	5	25	50	75	100
2,4,4'-Trichlorobiphenyl	2	5	25	50	75	100
3,3',4,4'-Tetrachlorobiphenyl	2	5	25	50	75	100
3,4,4',5-Tetrachlorobiphenyl	2	5	25	50	75	100
3,3',4,4',5-Pentachlorobiphenyl	2	5	25	50	75	100
3,3',4,4'5,5'-Hexachlorobiphenyl	2	5	25	50	75	100
AR1016		100	250	500	750	1000
AR1221				500		
AR1232				500		
AR1242				500		
AR1248				500		
AR1254				500		
AR1260		200	500	1000	1500	2000
AR5432		250	500	1000	2000	4000
AR5460		250	500	1000	2000	4000
AR6040				1000		
AR6062				1000		
AR6070				1000		
Tetrachloro-m-xylene		5	12.5	25.0	37.5	50.0
Decachlorobiphenyl		5	12.5	25.0	37.5	50.0
Hexabromobiphenyl		5	12.5	25.0	37.5	50.0

5.3 Surrogates. Tetrachloro-m-xylene, Decachlorobiphenyl and Hexabromobiphenyl are used in PCB/PCT analyses. Calibration levels are indicated on Table 2.

5.4 Calibration

If samples are to be analyzed for PCBs.

5.4.1 Five-level calibration of a mixture of Aroclor 1016 and Aroclor 1260 are initially analyzed. Percent RSD (Relative Standard Deviation) must be ≤20% for the Aroclors except surrogates. Analysis of single point mid level concentration of other five Aroclor listed in Table 1 are required. If PCB's are detected in the sample, patterns are compared for fingerprint match with initial standards and corresponding PCB's and retention time. Calculation is based on average area quantification of five representative peaks. When interferences are present, those peaks with less interferences may be chosen for quantification. Fewer peaks (minimum of 3 peaks) may be used for quantitation if there are considerable interferences.

5.4.2 Daily continuing calibration: Analyst should alternate the use of high and low concentrations of Aroclor 1016 and Aroclor 1260 at the beginning of each shift. Percent difference should be kept at ±5% for the Aroclors except for surrogates. If this criteria is exceeded for any Aroclor, calculate the average percent difference for all analytes in the calibration. And if the calculated average response factor for all analyte is within 15%, then the calibration is considered valid.

5.4.3 Mid sequence standard: mid-level mixture of Aroclor 1016/1260 should be injected every ten injections of samples and/or QC and at the end of every 12 hours, whichever is more frequent. All samples that were injected after the standard exceed the criteria of linearity must be re-injected if analyses indicate the presence of specific target analyte. However, if continuing calibration is ≥15% and no target analyte was detected, re-injection of the samples is not necessary. If continuing calibration ≤15% and no target analyte was detected, re-injection is necessary. Experience of the chromatographer is an influential factor in the determination of sample reanalysis.

If samples are to be analyzed for PCTs.

5.4.4 Initially 5 levels of standards containing AR5460 are analyzed and calibration curves for each target analyte is established. Mid-level continuing calibrations are analyzed during the sequence run when PCTs are required. If PCTs are detected in the sample, patterns and retention times are compared with the calibration standard. Calculation is based on average area or peak height of 5 representative peaks. When interferences are present, those peaks with less interferences may be

chosen for quantification. Also fewer peaks (minimum of 3) may be used for quantification if there are considerable interferences. Surrogate

Decachlorobiphenyl (DCB) and Tetrachloro-m-xylene (TCMX) are used for quality control monitoring. Acid clean up can be performed if matrix effects are observed. However, only one surrogate need to be calculated for recovery.

If samples are to be analyzed for congeners.

5.4.5 Initially five point calibration of PCB congeners are analyzed. The mean of the RSD value for all congeners must be equal or less than 20%. The initial calibration must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. A calibration standard must also be injected at the interval of 10 samples or/and QC samples, and at the end of the analysis sequence.

$$\% \text{ Difference} = \frac{CF_I - CF}{CF_I} * 100$$

Where:

CF_I = mean calibration factor from the initial calibration
CF = calibration factor from calibration verification standard

The calibration factor for each analyte calculated must not exceed a difference of more than 15% when compared to the mean calibration factor from the initial calibration. When calibration verification standard failed to meet the QC criteria, all samples that were injected after the last standard that met the QC criteria must be re-injected. However, if the sensitivity of the instrument had increase and target compounds are not present in the sample, there is no need for re-analysis

5.4.6 If samples are to be analyzed for low level congeners:

- The lowest calibration level shall be 2.0 ug/L.
- During sample preparation use 30 grams of sample with a final volume 2.0 ml
- The reporting limit shall be 0.1ug/kg.

5.4.7 When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of manual integration and notation of date and initials of person performing the task.

6.0 SAMPLE COLLECTION, HANDLING, PRESERVATION AND HOLDING TIMES

6.1 Sample extracts are preserved by keeping them cool to 4°C.

6.2 The holding times for extraction of the samples are 7 days of sampling for water samples and 14 days of sampling for soil samples. If sample has been extracted outside the holding time, note the aberration on the sample non-conformity report, and notify the manager for further instruction.

6.3 Sample must be analyzed within 40 days of the extraction. If sample has been analyzed outside the holding time, note the aberration on the sample non-conformity report, and notify the manager.

7.0 METHOD DETECTION LIMITS

7.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

8.0 METHOD PERFORMANCE

8.1 The MDL concentrations listed in the GPL MDL table are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

9.0 PROCEDURE

9.1 Retention time windows

9.1.1 Make 3 injections of midpoint standard mixtures throughout the course of a 72-hour period. Calculate standard deviation of the three absolute retention times for each single component standard. For multi-response products, choose one major peak from the cluster and calculate the standard deviation of the three retention times for that peak.

9.1.1.1 Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define retention time window. For multipeak products, primarily combination of chromatography pattern and retention times are used.

9.1.1.2 In those cases where the standard deviation for a particular standard is zero, substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

9.1.1.3 When a new GC column is installed retention time window must be established.

9.2 Sample Preparation

For sample preparation of soil and water, refer to GPL SOPs N.6 and N.7.

9.3 When all calibration requirements on both columns are met, sample analysis may begin. After each batch of 10 runs linearity should be checked before any more samples are analyzed. All samples that were injected after the standard exceeding the criteria must be re-injected, if the initial analysis indicates the presence of a specific Aroclor that exceeded the criteria.

9.4 Dilution must be made if the response exceeds the linear range of the compounds.

9.5 Peak identification is primarily based on detection on both columns within the established retention time. When results are confirmed using two dissimilar columns, the agreement between the quantitative results should be evaluated after identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula below.

$$RPD = \frac{|R1 - R2|}{(R1+R2)/2} \times 100$$

where R1 and R2 are the results on the two columns and the vertical bars in the equation above indicate the absolute value of the difference. Therefore, the RPD is always a positive value.

9.5.1 If one result is significantly higher (e.g., > 40%), check the chromatogram for anomalies. If there is no evidence of chromatographic problem, report the higher result with a J flag (for DOD project) and noted in the case narrative. The experience of the analyst may prove invaluable in determining whether the results are confirmed or not based on retention times and agreement between quantitation results of both columns.

9.6 GCMS Confirmation may be implemented if the concentration permits.

9.7 Wipe samples are treated like a solid sample and results are reported as ug per wipe. All QC parameters used for soil samples are applicable to wipes except for MS/MSD analysis. An actual MS/MSD analysis is impractical since only one wipe is sampled at a location and it cannot be split.

9.8 If peak detection and pattern identification are prevented by interferences, the extract should undergo acid cleanup using Method 3665A or sulfur cleanup using Method 3660B. The experience of the analyst may prove invaluable in determining which cleanup method is appropriate for particular samples.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 All quantifications are based on external standard calculations.

10.1.1 Calculation for calibration factor

$$\text{Calibration factor} = \frac{\text{Total Average Area of Peak*}}{\text{Mass injected (nonograms)}}$$

* for multipeak analytes use total areas of all designated peaks for quantitation

10.1.2 Percent Difference

$$\text{Percent difference} = \frac{(R_1 - R_2)}{R_1} \times 100$$

R_1 = Calibration factor from first analysis
R_2 = Calibration factor from succeeding analyses

10.1.3 The concentration of each Aroclor in the sample may be determined by calculating the amount of standard injected from the peak response, using the calibration factor determined from the initial five point calibration for Aroclor 1016 and for 1260. For other Aroclor that may be present in the sample, CF from the single point calibration standard of the specific Aroclor will be used for calculation.

Aqueous Concentration (ug/L) = $[(A_x)(A)(Vt)(D)]/[(As)(Vi)(Vs)]$

Solid Concentration (ug/kg) = $[(A_x)(D)(A)(V_t)]/[(As)(Ws)(Vi)]$

where:

A_x = Response for the analyte in the sample, (area or peak height
A = Amount of standard injected in ng
As = Response for the external standard
Vi = Volume of extract injected
D = Dilution factor, if any
Vt = Volume of total extract
Vs = Volume of sample extracted
s = Weight of sample extracted

For non-aqueous samples, the unit is µg/kg and dry weight of sample is used for Ws.

11.0 QUALITY CONTROL

11.1 Required Instrument QC

11.1.1 Percent RSD should be ≤20% when 5-point calibration factors are compared.

11.1.2 Percent difference of daily response of a given analyte should be within ±15% when compared to initial responses.

11.1.3 All succeeding standards in an analysis sequence must fall within daily retention time window established by the first standard of the sequence.

11.1.4 Control limits for MS/MSD, LCS, surrogates are established semi-annually for internal use.

11.1.5 For DOD projects, LCS, MS/MSD limits are evaluated against the DOD QSM control limits policy.

11.2 Matrix Spikes

11.2.1 MS and MSD must be analyzed with each batch of up to 20 samples of the same matrix processed together. If less than twenty samples are analyzed per month, MS/MSD must be analyzed on per month basis. Percent recoveries and Relative Percent Difference (RPD) should be calculated as follows:

$$\text{Matrix Spike \% Recovery} = \frac{SSR-SR}{SA} \times 100$$

where:

SSR = Spike Sample Results
SR = Sample Results
SA = Spike Added from Spiking Mix

$$RPD = \frac{D1 - D2}{(D1 + D2)/2} \times 100$$

D1 = First Sample Value
D2 = Second Sample Value

If samples are analyzed for PCBs only, then matrix spikes must be analyzed for AR1016 and AR1260.

If samples are analyzed for PCTs only, then matrix spikes must be analyzed for AR5460.

The MS/MSD is evaluated by comparing the precision of target analytes to the recovery windows established. MS/MSD data evaluation is more complex than method blank or LCS data since MS/MSD measure matrix effect in addition to sample preparation and analysis error. MS/MSD that fail to meet the acceptance criteria would indicate that a potential matrix effect is present. The laboratory must assess the batch to determine whether the spike results are attributable to matrix affect, or the result of another problem in the analytical process. If all the QC batch elements, which are not affected by the sample matrix, are in control (e.g., method

blank, LCS), and if there is no evidence that the spiking was not properly performed, the poor spike recovery may be attributed to matrix effect. If the LCS compounds that are not affected by the sample matrix are out of control, and if the same compounds in the MS/MSD are outside control limit, then matrix spiked sample(s) must be re-processed through the entire analytical procedure.

11.3 Lab Control Sample (LCS)

11.3.1 A control check sample is extracted and analyzed at frequency similar to MS/MSD with each extracted batch. Lab Control Spikes (LCS) are analyzed per batch or every 20 samples, whichever comes first. LCS compound must be within the established control limit. Statistical control limit are based on at least 30 data points. Data points used in the data set must not be selectively included or excluded. If recovery of LCS compound falls outside the established limit, corrective action must be taken. After corrective action, if LCS analyte recovery is still outside QC acceptance limits, the entire associated samples batch must be re-extracted if holding times have not elapsed. When PCB analysis is needed the control check sample should be spiked with AR1016 and 1260 at the same levels as matrix spikes. When PCT analysis is needed the control check sample should be spiked with AR5460 at the same levels as matrix spikes.

11.4 Blanks

11.4.1 Blank is extracted and analyzed with each analytical batch. Blanks must be contaminant free. Concentration of any confirmed peak should be less than one half of the reporting limit.

11.5 Surrogates

11.5.1 Percent recoveries for the surrogates are determined by plotting percent recoveries of surrogates measured in 20 consecutive blanks. Control limits for each surrogate compound is measured using the following formula:

$$\text{Upper Control Limit (UCL)} = p + 3s$$
$$\text{Lower Control Limit (LCL)} = p - 3s$$

where p is the mean recovery and s is the standard deviation.

11.5.2 Two surrogate (TCMX and DCB) are added to each sample, however, only one need to be calculated for recovery. Calculate surrogate standard recovery on all samples, blanks and spikes. Determine if the recovery is within limits. If recovery is not within limit, re-extract and reanalyze the sample.

11.5.3 For DOD projects, surrogate limits are evaluated against the DOD QSM control limits policy.

12.0 SAFETY

12.1 Safety glasses for eye protection, laboratory coasts for body protection, latex gloves for hand protection.

12.2 Due to the toxicity or carcinogenicity of each reagent, each chemical compound should be treated as a potential health hazard.

12.3 Material Safety Data Sheets (MSDS) can be found on the procurement bookshelves located in the company library.

12.4 Preparation of the standard should be handled under a hood.

13.0 POLLUTION PREVENTION

13.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

14.0 WASTE MANAGEMENT

14.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

15.0 DEFINITIONS

15.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

16.0 REFERENCES

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition.

- DOD Quality System Manual , Final Version 3, January 2006

SOP No: Q.10

Title: Analysis of Herbicides by Method 8151A

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the determination of certain chlorophenoxy acid herbicides by GC/ECD.

1.2 The following compounds can be determined using this method:

2,4-D	2,4,5-T	2,4,5-TP
2,4-DB	Dichloroprop	MCPP
Dalapon	Dinoseb	4-Nitrophenol
Dicamba	MCPA	Pentachlorophenol

2.0 SUMMARY OF METHOD

2.1 Method 8151A provides extraction, esterification, and gas chromatographic conditions for the analysis of chlorinated phenoxy acid herbicides. The esters are hydrolyzed with potassium hydroxide, and extraneous organic material is removed by a solvent wash. After acidification, the acids are extracted with solvent and converted to their methyl esters using diazomethane as the derivatizing agent. After excess reagent is removed, the esters are determined by gas chromatography employing an electron capture detector. The results are reported as the acid equivalents.

3.0 INTERFERENCES

3.1 Organic acids, especially chlorinated acids, cause the most direct interference with the determination. Phenols, including chlorophenols, may also interfere with this procedure.

3.2 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis.

3.3 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must, therefore, be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

3.4 Interference's co-extracted from the samples will vary considerably from waste to waste. While general cleanup techniques are provided as part of this method, unique samples may require additional cleanup approaches to achieve desired sensitivities.

3.5 Glassware must be scrupulously clean. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing in hot water. Rinse with tap water, distilled water, acetone, and finally pesticide-quality hexane. Heavily contaminated glassware may require treatment in a muffle furnace at 400°C for 15 to 30 minutes.

4.0 SAFETY

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis.

5.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

5.1 Samples are preserved by keeping them cooled to 4°C.

5.2 The holding time for extraction of the water samples is 7 days of sampling and 14 days for soil samples.

6.0 APPARATUS AND MATERIALS

6.1 Gas Chromatograph

6.1.1 Gas Chromatograph

Analytical system complete with gas chromatograph suitable for splitless mode capillary column injections and all required accessories, including detector, column supplies, recorder, gases and syringes. A data system for measuring peak heights. The gas chromatograph is a Hewlett Packard 5890 Series II.

6.1.2 Columns - Dual column connected with a "Y" connector to a guard column is used. In this mode, material injected is split between the columns and detected by 2 separate ECD detectors.

6.2 Vials: Amber glass, 10 to 15mL capacity with Teflon-lined screw cap

6.3 Microsyringe: 10uL

6.4 Syringe: 5mL

7.0 REAGENTS AND STANDARDS

7.1 Solvents: Acetone, methanol, ether, methylene chloride, hexane (pesticide quality or equivalent).

7.2 Stock standard solutions: Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

- Prepare stock standard solutions by accurately weighing about 0.0100g of pure methylesters. Dissolve the material in hexane (pesticide quality) solvent and dilute to volume in a 10mL volumetric flask. Larger volumes can be used at the convenience of the analyst. If compound purity is certified at 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

- Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

- Stock standard solutions must be replaced after 1 year, or sooner if comparison with check standards indicates a problem.

8.0 CALIBRATION AND STANDARDIZATION

8.1 Calibration standards at a minimum of five concentration levels (listed below) for each parameter of interest are prepared through dilution of the stock standards with hexane. One of the concentration levels should be at a concentration near or below the reporting limit. The remaining concentration levels correspond to the expected range of concentrations found in the real samples or define the working range of the GC. Calibration solutions must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

Methyl Ester Concentrations in µg/L (ppb)

Compound	I	II	III	IV	V
2,4-DCAA (Sur)	100	200	300	400	500
2,4-D	100	200	300	400	500
2,4-DB	100	200	300	400	500
2,4,5-TP (Silvex)	100	200	300	400	500
2,4,5-T	100	200	300	400	500
Dalapon	100	200	300	400	500
Dicamba	100	200	300	400	500
Dichloroprob	100	200	300	400	500
Dinoseb	100	200	300	400	500
MCPA*	10	20	30	40	50
MCPP*	10	20	30	40	50
4-Nitrophenol	100	200	300	400	500
Pentachlorophenol	100	200	300	400	500

* Conc. µg/mL (ppm)

8.2 Surrogate Standards

Compound 2,4-dichlorophenyl acetic acid has been chosen to be the surrogate for this analysis. See above calibration table for actual calibration ranges. Methyl ester of this compound is used in calibration, where 0.5mL of 10ug/ml of the acid form of this compound is added to sample and QC.

Table 1
Lists the Conversion Factors for Calibrating the Instrument
for Calculation of Acid Equivalents

	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5
Dalapon-ME	109.8	219.6	329.4	439.3	549.1
Dicamba-ME	106.3	212.7	319.0	425.4	531.7
MCPA-ME	10699	21399	32098	42797	53497
Dichloroprop-ME	106.0	211.9	317.9	423.9	529.8
2,4-D-ME	106.3	212.7	319.0	425.4	531.7
MCPP-ME	10699	21399	32098	42797	53497
2,4,5-TP (Silvex)-ME	105.2	210.4	315.6	420.8	526.0
2,4,5-T-ME	105.5	211.0	316.5	421.9	527.4
2,4-DB-ME	94.4	188.7	283.1	377.5	471.8
Dinoseb-ME	105.8	211.7	317.5	423.3	529.2
Pentachlorophenol-ME	105.3	210.5	315.8	421.1	526.3
4-Nitrophenol-ME	110.1	220.2	330.2	440.3	550.4
2,4-DCAA-ME	106.8	213.7	320.5	427.4	534.2

9.0 CALIBRATION

9.1 Inject 2.5 microliter of each calibration standard using the splitless mode. Tabulate peak height or area responses against the mass injected. The results are used to prepare a calibration curve for each analyte. Alternatively, the ratio of the response to the amount injected, defined as the calibration factor (CF), can be calculated for each analyte at each standard concentration. If the percent relative standard deviation (%RSD) of the calibration factor is less than $\leq$ 20% over the working range, linearity through the origin can be assumed, and the average calibration factor can be used in place of the calibration curve.

$$\text{Calibration factor} = \frac{\text{(Area or Height) of Peaks}}{\text{mass injected (nanograms)}}$$

9.2 The working calibration curve or calibration factor must be verified on each working day by the injection of one calibration standards. Continuing calibration standard must be analyzed every 12 hours. Analyst should alternate the use of high and low concentration for calibration verification. If the response for any analyte varies from the predicted response by more than $\pm$ 15%, corrective action is required. After corrective action if criterias are not met a new calibration curve must be prepared for that analyte.

$$\text{Percent Difference} = \frac{[R_1 - R_2]}{R_1} \times 100$$

where:

R_1 = Average Calibration Factor from the Initial Calibration
R_2 = Calibration Factor from Continuing Calibration Standard

The calibration standard must be verified between every ten injections and at the end of every 12 hours, whichever is more frequent. All samples that were injected after the standard exceeding the criteria of linearity ($\pm$ 15%) must be re-injected.

9.3 Retention Time Windows

9.3.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of all calibration standard mixtures.

9.3.2 Calculate the standard deviation of the three absolute retention times for each component.

9.3.3 The mean retention time plus or minus three times the standard deviation of the mean retention time for each component will define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

9.3.4 In those cases where the standard deviation for particular standard is zero, the laboratory must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

9.3.5 The retention time windows are updated at least weekly or upon sample analysis if the method is performed less frequently.

9.3.6 The laboratory must calculate retention time windows for each component on each GC column and whenever a new GC column is installed. Table 3 lists typical retention times for target herbicides for primary and confirmation columns.

10.0 METHOD DETECTION LIMITS

10.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table

11.0 METHOD PERFORMANCE

11.1 The MDL concentrations listed in the GPL MDL table are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and a accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

12.0 PROCEDURE

Extraction of soil and water samples, refer to GPL SOP N.17.

12.1 Before processing any samples, the analyst should demonstrate through the analysis of a reagent water blank, that interferences from the analytical system, glassware and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent water blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement steps.

12.2 For each analytical batch (up to 20 samples), a reagent blank, a spiked blank, matrix spike and matrix spike duplicate must be analyzed. The blank, spiked blank and spiked samples must be carried through all stages of the sample preparation and measurement steps.

13.0 QUALITY CONTROL

13.1 Initial calibration requires that a %RSD vary by ≤20% when comparing calibration factors to determine if a five point calibration curve is linear.

13.2 The daily continuing calibration sets a limit of ± 15% difference when comparing daily response of a given analyte versus the initial response. If the limit is exceeded, a new standard curve must be prepared.

13.3 All other continuing calibrations sets a limit of ± 15% difference when comparing the initial response of a given analyte versus any succeeding standards analyzed during an analysis sequence.

13.4 A method blank is extracted and analyzed with each preparation batch. If the method blank contains any analytes of interest above one half of the reporting limit, the batch is rejected and the samples must be re-extracted and re-analyzed if sufficient sample exists and holding times have not elapsed.

13.4 The laboratory must, on an ongoing basis, perform at least one Matrix Spike/Matrix Spike Duplicate per analytical batch (maximum of 20 samples per batch) to assess accuracy. If ten or less samples are analyzed within a given month, at least one MS/MSD sample per 14 days is required. At least one matrix spike (no duplicate) is extracted and analyzed per each TCLP batch. Spike with all the compounds of interest as required.

13.5 To determine acceptable accuracy and precision limits for surrogate standards, the following procedure should be performed:

13.5.1 For each blank analyzed, calculate the percent recovery of each surrogate in the blank.

13.5.2 Once a minimum of 20 blanks of the same matrix have been analyzed, calculate the average percent recovery (p) and standard deviation of the percent recovery (s) for each of the surrogates.

13.5.3 For a given matrix, calculate the upper and lower control limit for method performance for each surrogate standard. This should be done as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

where p is the mean and s is standard deviation

13.5.4 If recovery is not within limits, the following is required:

- Check to be sure there are no errors in calculations, surrogate solutions. Also, check instrument performance and recalculate data.

- If surrogate recovery in the method blank fails the QC criteria, refer to the lab supervisor for evaluation of data, if re-extraction is necessary.

13.6 The laboratory performs, on an ongoing basis, one blank spike (LCS) per analytical batch (maximum of 20 samples per batch). If fewer samples are analyzed within a given month, at least one LCS per 14 days is required.

13.6.1 When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

13.7 Summary of QC

Surrogates	Every sample and standard
Calibration check standard	Within 12 hours
Method blank	One per batch, per matrix
Matrix spike	One per batch, per matrix
Matrix spike duplicate	One per batch, per matrix
QC blank spike sample (Laboratory Control Sample)	One per batch, per matrix

13.8 Control limits for MS/MSD, LCS and surrogates are established semi-annually for internal use.

14.0 INSTRUMENTAL PROCEDURES

14.1 Gas Chromatography

14.1.1 Gas Chromatography Conditions

Column 1: RTX-CLP

Carrier Gas	Helium
Split Flow	60ml/min
Purge Flow	3ml/min
Column Flow	4ml/min @ 180°C
Make-Up Flow	60ml/min (95:5 Argon/Methane)
Injector Port	200°C
Detector Temp	300°C
Column Temp	60°C
Ramp 1 Rate	25°C
Final Temp	100°C
Hold	4.4 min.
Ramp 2 Rate	30°C
Final Temp	300°C
Hold	5.0 min.

Split Valve On	1.0 min.
Split Valve Off	0.0 min.

Column 2: RTX-CLP2

Carrier Gas	He
Split Flow	60ml/min
Purge Flow	3ml/min
Column Flow	4ml/min @ 180°C
Make-Up Flow	60ml/min (95:5 Argon/Methane)
Injector Port	200°C
Detector Temp	300°C
Column Temp	60°C
Ramp 1 Rate	25°C
Final Temp	100°C
Hold	4.4 min.
Ramp 2 Rate	30°C
Final Temp	300°C
Hold	5.0 min
Split Valve On	1 min
Split Valve Off	0 min

14.2 Suggested chromatography system maintenance: corrective action may require the following remedial action.

14.2.1 Capillary columns: Clean and deactivate the glass injection port insert or replace with a cleaned deactivated insert. Carefully cut off the first few inches (up to one foot) of the injection port side of the column. Remove the column and solvent backflush according to the manufacture's instructions. If degradation problems persist, replace the column.

14.3 A compound is qualitatively identified as being present if the peak is detected within the established retention time window for that specific compound. To confirm that an analyte is present, the extract must be run on a secondary column. Again, the peak must be detected within the established retention time window.

14.4 Peak identification is primarily based on detection on both columns within the established retention time. When results are confirmed using two dissimilar columns, the agreement between the quantitative results should be evaluated after identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula below.

$$RPD = \frac{|R1 - R2|}{(R1+R2)/2} \times 100$$

where R1 and R2 are the results on the two columns and the vertical bars in the equation above indicate the absolute value of the difference. Therefore, the RPD is always a positive value.

14.4.1 If one result is significantly higher (e.g., > 40%), check the chromatogram for anomalies. If there is no evidence of chromatographic problem, report the higher result and noted in the case narrative. The experience of the analyst may prove invaluable in determining whether the results are confirmed or not based on retention times and agreement between quantitation results of both columns.

14.5 The target analytes concentration must be within the calibration range for quantitation. If the sample requires dilution, the dilution must also be acquired on a secondary column for confirmation.

DATA ANALYSIS AND CALCULATIONS

External Standard Calibration

Aqueous Samples:

$$\text{Concentration (ug/L)} = [(A_x)(V_t)(D)]/[(CF)(V_f)(V_s)]$$

Where:

A_x = Response for the analyte in the sample, units are peak heights or area counts

CF = Calibration Factor (See Section 8.1)

V_i = Volume of extract injected, uL

D = Dilution factor - if dilution was made on the sample prior to analysis. If no dilution was made, D = 1, dimensionless

V_t = Volume of total extract, uL

V_s = Volume of sample extracted, ml

Nonaqueous Samples:

$$\text{Concentration (ug/kg)} = [(A_x)(D)(V_t)]/[(CF)(W_s)(V_i)]$$

Where:

A_x = Response for the analyte in the sample, units are peak Heights or area counts

CF = Calibration Factor (See Section 8.1)

V_i = Volume of extract injected, uL

D = Dilution factor - if dilution was made on the sample prior to analysis. If no dilution was made, D = 1, dimensionless

V_t = Volume of total extract, uL

W_s = Weight of sample extracted. Either dry weight or wet weight may be used, depending upon the specific application. All concentrations are to be reported on a dry weight basis otherwise specified.

14.6 Percent Recovery (%R)

$$\%R = \frac{\text{(Observed - Sample) Concentration}}{\text{Expected Concentration}} \times 100$$

14.7 Relative Percent Difference (RPD)

$$RPD = \frac{\text{(\%Measurement 1 - \%Measurement 2)}}{\text{Average of Measurement 1 and 2 (\%)}} \times 100$$

15.0 REPORTING OF RESULTS

15.1 Aqueous results are routinely reported as ug/L and nonaqueous results are ug/kg.

15.2 TCLP results are to be reported as μg/L.

15.3 The number of significant figures to be reported is 2.

15.4 If the analyte is detected above the reporting limits, the value will be reported.

15.5 If there is no evidence of the presence of the analyte, the analyte is reported as ND.

15.6 If the analyte is detected below the reporting limit, the analyte is reported as (J) value.

16.0 POLLUTION PREVENTION

16.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

17.0 WASTE MANAGEMENT

17.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

18.0 DEFINITIONS

18.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

19.0 REFERENCES

- U.S. EPA 40 CFR Part 136: Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act. October 1984.

- Test Methods for Evaluating Solid Waste. United States Environmental Protection Agency, SW-846, Volume 1-B Laboratory Manual, Third Edition, December1996, "Method 8000B Gas Chromatography" and "Method 8151A Chlorinated Herbicides".

SOP No: P.5

TITLE: SOP for Method SW8270C
GC/MS Analysis of Semivolatile Organics

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure describes the methodology used to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and water. Table 1 indicates compounds typically determined by this method and lists the method reporting limit for each in water or soil. Table 2 lists the Appendix IX compounds which can be determined using 8270C methodologies. Table 3 also lists additional compounds that can be determined using Method 8270C. Table 4 lists the semivolatile PAH compounds analyzed by low concentration.

2.0 SUMMARY OF METHOD

2.1 Prior to using this method, the samples should be prepared for chromatography using the appropriate sample preparation methods.

3.0 INTERFERENCE

3.1 GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples and initiate corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between each use.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph/Mass Spectrometer

Hewlett Packard 5970 and 5972 MSD System equipped with an autosampler is utilized. The system is a complete instrument composed of two modules: a Controller and a main frame GCMS.

4.1.1 Data System

Each GC/MS system is attached to a dedicated 486 or pentium based computer equipped with Enviroquant software for automated acquisition and processing. The software generates total and extracted ion profiles of each compound and is capable of performing library searches on spectra using a full EPA/NIH Mass Spectral Data Library. Each system is attached to an internal laboratory computer network for additional data processing, storage and archiving.

4.2 Columns

A 30 meter, DB-5ms or equivalent (5% phenyl methyl silicon) column is used.

4.3 Syringe: 10µl

5.0 GC CONDITION

5.1 The following GC parameters are suggested:

-	Injection port temp:	250°C
-	Interface temp:	300°C
-	Initial temp:	40°C
-	Initial time:	4 min.
-	Rate at	15°C per min.
-	Final temp:	310°C
-	Final time:	20 min.
-	Equilibrium time:	.5 min.
-	Septum purge flow on at	.5ml/min.
-	Septum purge flow on at	.5 min.
-	Flow rate at about	0.7ml/min.
-	Splitless flow about	50ml/min.
-	Injection type:	"Splitless"
-	Autosampler injection mode:	"Fast"
-	Sample Volume	1µl

5.2 GC/MS Condition

5.2.1 Scanning from 35 to 500amu in less than 1 second, using 70 volt (nominal) electron energy in the Electron Impact Ionization mode.

5.2.2 GCMS Tuning - to determine the system performance, 50ng of decafluorotriphenylphosphine (DFTPP) is injected and the mass spectrum is evaluated. Verify that the MS meets standard mass spectral abundance criteria. The tune standard must be analyzed at the beginning of each analytical shift and every 12 hours of continuous analysis. Evaluate the ion abundances using any of the following:

- Use one scan at the apex;
- Use one scan either directly preceding or following the apex;
- Use the mean of the apex and the preceding and following scans;
- Use the average across the entire peak.

Background correction should be employed only for the purpose of correcting for instrument background ions. If any single approach fails, re-inject the DFTPP standard or retune the instrument. The following criteria must be met before any further analysis is performed.

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

MASS	ION ABUNDANCE CRITERIA
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

The GC/MS tuning standard should also be used to access GC column performance and injection inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and Pentachlorophenol should be present at their normal responses, and no peak tailing should be visible. The acceptance criteria for the peak tailing factor for benzidine is <3.0 and pentachlorophenol is <5.0. For samples received from the State of California, the following documentation will be provided: percent breakdown for DDT and tailing factors for benzidine and pentachlorophenol. The calculation of peak tailing factors is illustrated in Figure 1.

5.3 Calibration

5.3.1 Upon satisfactory completion of DFTPP analysis, six levels of calibration standards at 10,20, 50, 80, 120 and 160ng are analyzed. Calibration standards must contain the following internal and surrogate standards.

5.3.1.1 Internal Standards - the following Internal Standards at 40mg/ml must be present in the Calibration Standard.

- 1,4-dichlorobenzene-d_4
- Naphthalene-d_8
- Acenaphthane-d_{10}
- Phenanthrene-d_{10}
- Chrysene-d_{12}
- Perylene-d_{12}

5.3.1.2 The following Surrogate Standards at 10,20, 50, 80, 120 and 160ng are analyzed.

- Phenol-d_6
- 2-Fluorophenol
- 2,4,6-Tribromophenol
- nitrobenzene-d_5
- 2-fluorobiphenyl
- p-terphenyl-d_{14}

5.4 After analyzing the initial Calibration Standard average RF should be calculated for each compound. The percent relative standard deviation (%RSD) should be equal or less than 15% for each compound. However, Calibration Check Compounds (CCC) must have a % RSD less than or equal to 30%. It is likely that some analytes may exceed the 15% acceptance limit for the %RSD. In those instances the following steps should be considered.

- If the %RSD is greater than 15%, the analyst should review the results (area counts, response factors, standard concentrations) for those analytes to ensure that the problem is not associated with a single standard. If so, that standard should be reanalyzed and RSD recalculated. Replacing of standard may be necessary in some cases.

- It may also be necessary to narrow the calibration ranges to achieve a better linearity. The changes to the upper end of the calibration will affect the need to dilute samples above that range, while the change to the lower calibration will affect the sensitivity and elevate the reporting limit of the method. When considering dropping one or two points to narrow the calibration range, a minimum of five points is required.

After visual inspection of the calibration points for each analytes and for those analytes that did not meet the %RSD criteria, at the discretion of the analyst it should consider to employ a linear regression that does not through the origin or a quadratic (second order) model. A quadratic model requires six standards. Linearity is presumed acceptable if the correlation coefficient (r) is equal to or greater than 0.995 or the coefficient of determination (r^2) is equal to or greater than 0.99.

The relative retention times of each compound in each calibration run should agree within 0.06 relative time units. System performance check compounds must have a minimum RF of 0.05. All other compounds must have minimum RF of 0.01. The CCC and SPCC compounds are listed as follows:

SPCC	CCC
N-nitroso-di-n-Propylamine	Acenaphthene
Hexachlorocyclopentadiene	1,4-Dichlorobenzene
2,4-Dinitrophenol	N-Nitrosodiphenylamine
4-Nitrophenol	Di-n-octylphthalate
	Fluoranthane
	Benzo(a)pyrene
	4-Chloro-3-methylphenol
	2,4-Dichlorophenol
	2-Nitrophenol
	Phenol
	Pentachlorophenol
	2,4,6-Trichlorophenol

5.5 Prior to use for sample analysis, the acceptability of initial calibration curve must be verified through analysis of a calibration verification solution obtained from a second source. Calibration verification must meet the same acceptance criteria used for continuing calibration (daily) outlined in Section 5.7.

5.6 Internal Standard responses and retention times in the calibration standard, blanks and samples must be evaluated. Retention time variation for any internal standard in the calibration standard must be less than 30 seconds from that in the midpoint standard level of the most recent initial calibration. All other samples and QC samples must be compared to the daily CCV. Variation of the areas for internal standards must not vary by more than a factor of two or -50% to +100%.

5.7 A continuing calibration (daily) at 50ng concentration containing all semivolatile analytes, including all required surrogates must be analyzed every 12 hours. The following criteria must be met before sample analysis starts:

- System Performance Check Compounds (SPCCs): A system performance check must be made every 12 hour shift. For each SPCC compound in the daily calibration a minimum response factor of 0.05 must be obtained.

- All other non SPCC must have RF of 0.01.

- Percent difference for all CCC should be less than 20%.
  Non CCC should have less than 20% except up to 8 poor performing compounds could have 40%. If % drift for any poor performing compound is greater than -35% (lost sensitivity)and that compound is present in the sample, the sample should be re-analyzed with acceptable calibration standard.

For DOD projects, non CCCs shall meet less than 25%D criteria.

$$\%\text{Difference} = \frac{RRF_c - \overline{RRF_i}}{RRF_i} \times 100$$

where:

RRF_c = Relative response factor from continuing calibration standard

RRF_i = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

If the % difference criteria for each CCC and non CCC is not met corrective action must be taken, and if after corrective action criteria were not met, a new initial calibration must be generated.

- Retention time for Internal Standards should not drift more than ± 30 seconds compared from that in the mid point standard of the most recent initial calibration.

6.0 METHOD DETECTION LIMITS

6.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

7.0 METHOD PERFORMANCE

7.1 The MDL concentrations listed in the GPL MDL/RL table are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

8.0 SAMPLE ANALYSIS

8.1 To 1ml of BNA extracts, 20uL of 2000mg/ml internal standard is added to each mix. One ul of this extract is injected into the GCMS using the same parameters as the calibration standard. All samples must be injected within a 12 hour period starting with analysis of DFTPP.

8.2 Qualitative Analysis

8.2.1 Two criteria must be satisfied to verify compound identification.

8.2.1.1 Elution of sample component at the same GC relative retention time (RRT) ± 0.06 units.

8.2.1.2 Correspondence of the sample component and the standard component mass spectrum.

All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum. The relative intensity of these ions must agree ± 20% between the standard and sample spectra.

8.2.2 Library Search

8.2.2.1 When required, a search may be performed for the purpose of tentative identification. Only after visual comparison of sample spectra with the nearest library searches will the analyst assign a tentative identification. The analyst should use an approach similar to the 5 step identification listed for method SW8270 (pp-20), Dec. 1996.

8.3 Quantitative Analysis

8.3.1 When a compound is identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation is based on the internal standard technique. The internal standard nearest the retention time of that given analyte, shall be used.

9.0 CALCULATIONS

The following information should be used throughout the quantitation process.

9.1 Response Factor

$$RF = \frac{(A_x C_{is})}{(A_{is} C_x)}$$

where:

A_x = Area of the characteristic ion for the compound being measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_x = Concentration of the compound being measured (mg/ml)

C_{is} = Concentration of the specific internal standard (mg/ml)

9.2 % RSD and % Difference

$$\% \text{ RSD} = \frac{\text{Standard Deviation}}{\text{Average RF}} \times 100$$

$$\% \text{ Difference} = \frac{\text{AvgRF}_i\text{(Init. Calib.)} - \text{RF}_c\text{(Cont. Calib.)}}{\text{Avg RF}_1} \times 100$$

9.3 Sample Calculation

$$\text{Water Concentration ug/l} = \frac{(A_x)\,(I_s)\,(V_t)\,(\text{Dil})}{(A_{is})\,(\text{Avg RF})\,(V_o)\,(V_i)}$$

$$\text{Soil Concentration ug/kg} = \frac{(A_x)\,(I_s)\,(V_t)\,(\text{Dil})}{(A_{is})\,(\text{Avg RF})\,(V_i)\,(W_s)\,(D)}$$

where:

I_s = Amount of internal standard injected (ng)

V_t = Volume of total extract

V_o = Initial volume

V_i = Volume of extract injected (ml)

W_s = Weight of sample extracted (G)

D = Percent solids

Dil = Dilution applied

Alternatively, the regression line fitted to the initial calibration may be used for determination of the extract concentration.

10.0 QUALITY CONTROL

10.1 Blank analysis - A method blank is analyzed with each analytical batch to examine the interferences and contamination from extraction and analysis. Concentration of the target compounds in the blank must be less than or equal to ½ of the reporting limit. If the above criteria are not met, re-injection or re-extraction of the blank and the samples associated with the blank if sufficient sample exists and holding times have not elapsed.

10.2 Surrogate analysis - Surrogate standard determinations are performed on all samples and blanks.

For DOD projects, the QSM control limits policy shall be followed. First, the project-specific limits will be used if available. If not, QSM limits and policy shall be used, if available. Other wise, the internal limits shall be used.

Policy for non-DOD projects:

10.2.1 If any surrogate recoveries in the method blank in either the BN or acid fraction are outside the surrogate spike recovery limits, corrective action should be taken. First, re-inject the blank - if no improvement is observed, reextraction should be considered. The blank and all samples associated with the blank should be re-extracted if enough sample exists and holding time have not elapsed.

10.2.2 If more than one BN or acid fraction in the sample are outside the surrogate spike recovery limits, corrective action should be taken. First, re-inject the sample. If the surrogate recoveries fail to meet the criteria after re-analysis, re-extract the sample. If the surrogate compound recoveries meet acceptance criteria in the re-extraction/reanalysis, submit data from the re-extraction/reanalysis only if it met holding times. If not, submit both data.

10.2.3 If any surrogate in the sample has a recovery limit less than 10 percent, first re-analyze the sample. If sample does not meet recovery limit, re-extract and re-analyze the sample if enough sample exists and holding time have not elapsed.

10.2.4 If the surrogate compound recoveries fail to meet the acceptance criteria in the re-extraction/reanalysis sample, then submit data from both analyses.

10.2.5 If the surrogate recoveries in the sample have been affected by obvious matrix interferences and reanalysis of the extract may harm the analytical system, report the data and explain in the case narrative.

10.3 Matrix Spikes - A matrix spike and matrix spike duplicate are analyzed with each batch of up to 20 samples of the same matrix processed together. MS/MSD recoveries should be within the in-house established control limits. Control limits are established based on minimum 20-30 historical data points that span between 6 months to a year. The results from the MS/MSD normally would not be used to determine the validity of the entire batch. The poor performers in the spike may indicate a problem with sample composition and shall be reported to the client as such.

If matrix spike recovery does not meet the Control Limit criteria, the supervisor must asses the data to determine whether the spike results are attributed to a

matrix affect, or the results of other problem in the analytical process. If all QC elements, which are not affected by the sample matrix, are in control (e.g. Method Blank, LCS, Calibration check), and if there is no evidence the spiking may have been improperly performed, the poor spike recovery may be attribute to matrix affect. In this case corrective action is not required.

If any of the batch QC elements which are not affected by sample matrix are out of control, or if there is any evidence that spiking may have been improperly performed, the matrix spike sample must be reprocess. MS/MSD pair are spiked the same as the LCS components.

For DOD projects, DOD QSM matrix spike control limits policy shall be followed.

10.4 Lab Control Sample

10.4.1 A laboratory control sample is extracted and analyzed routinely with each extracted batch. Calculated concentrations are compared with the amount added and results are used to demonstrate that the laboratory process for sample preparation and analysis is in control. Generation of in house statistical control limits must be based on minimum 30 data points. Data set used to generate control limits must have been generated using the same analytical procedure and data sets must not be selectively included or excluded.

Each analyte in the LCS must be evaluated against the Control Limits and Marginal Exceedance limit. If any LCS is outside the Control Limit, it should be also compared to the Laboratory Marginal Exceedance Limits to ensure that it does not exceed. If a single analyte in the LCS exceeded the Marginal Exceedance Limit, the LCS had failed and corrective action needs to be initiated.

If the LCS has more than the allowable number of Marginal Exceedance, the LCS has failed and corrective action needs to be initiated.

The corrective action for failed LCS is based on professional judgment in conjunction with matrix spike and surrogate recoveries in the same batch. If after checking the associated QC's it was determined by the section supervisor or manager that the LCS had failed, all affected samples associated with the out of control LCS should be reprocessed and re-analyzed.

If samples cannot be reprocessed due to lack of sample volume or the holding times has lapsed, the results should be reported with appropriate flag. The report case narrative must include a discussion of the failed LCS, and its impact on the data quality.

When LCS is spiked with large number of analytes, the laboratory should add up total number of exceedances for the LCS based on the number of analyte spiked in the LCS. The total of exceedance should be compare with the allowable number from the following chart.

Number of analyte in the LCS	Allowable Number of Marginal Exceedances of LCS
< 11	0
11-30	1
31-50	2
51-70	3
71-90	4
>90	5

Since a large amount of GPL's clients most requested compound list are fairly long (> 20 compounds), the laboratory will spike all the reportable compounds as specified in the clients project. At a minimum, 16 compounds from the list will be selected and used to verify if the LCS meets the required QC requirements. However, the laboratory should ensure that all compounds in the target list are used to verify the LCS criteria over a period of two years.

10.5 Internal Standard Recoveries and Retention times - when internal standard recoveries (-50 to +100%) or retention time requirements, (0.06 RRT) are not satisfied, sample is reinjected and reanalyses results are submitted if they are acceptable. If both analyses are out of criteria, then both results are submitted.

10.6 Sample Collection and Preservation.

10.6.1 Water samples may be collected in 1L (or one quart) amber glass container. Soil samples may be collected in glass containers or closed end tubes.

10.6.2 All samples must be iced or refrigerated at 4°C (± 2°C) from the time of collection until extraction.

10.6.3 Extracts of water and soil/sediment samples must be analyzed within 40 days following extraction.

10.7 Dilutions are performed on samples when the concentrations of target analytes exceed the calibration range. Additional IS must be added to the diluted extract to maintain the required IS concentration. Dilution of extracts should result in analysis with the highest concentration target analyte in the upper half of the calibration range.

10.8 QC limits for MS/MSD, LCS and surrogates are established semi-annually for internal use.

11.0 LOW CONCENTRATION

11.1 When requested, low concentration can be analyzed using a modified 8270C method. Lower detection limit is achieved by analyzing low level standards at 2-4ul injection. Instrument is optimized for high masses and the multiplier is

enhanced to achieve the desired increased sensitivity. The instrument (the source) and injection port need to be scrupulously clean when analyzing low level semivolatile compounds. Sample matrix should be reasonably free of interferences to allow low level detection of semivolatile compounds. Extraction procedures for water and soil matrices are the same as methods 3510, 3520, 3540 and 3550 except for surrogate and matrix spike standards levels added to the sample and quality control samples before extraction. Surrogate standard is added at 1.0ml of 0.5ug/ml of all compounds. Matrix spike and lab control spike consist of 1.0ml of all compounds at 0.5ug/ml concentrations. Both soil and water matrices receive the same amount of surrogate and matrix spike standards. Sample and quality control extracts are spiked with 4.0ug/ml internal standard before injection.

11.2 Seven level calibration standards are analyzed at a concentration 0.1, 0.5, 1.0, 2.5, 5.0, 10, and 20ug/ml. All Method 8270C QC criteria's applies to the low level 8270 analysis.

12.0 MS SIM ANALYSIS

12.1 When requested, low concentration target analyte can be analyzed using Selective Ion Monitoring (SIM) mode. Selective Ion Monitored (SIM) mode is a data acquisition technique in which only a few selected ion fragments are monitored in order to obtain maximum selectivity. Extraction procedures for water and soil matrices are the same as methods 3510, 3520, 3540 and 3550 except for surrogate and matrix spike standards levels added to the sample and quality control samples before extraction. Surrogate standard is added at 1.0ml of 0.5ug/ml. Matrix spike and lab control spike consist of 1.0ml of all compounds at 0.5ug/ml concentrations. Both soil and water matrices receive the same amount of surrogate and matrix spike standards. Sample and quality control extracts are spiked at 5.0ug/ml internal standard before injection.

12.2 Seven level calibration standards are analyzed at a concentration 0.1, 0.5, 1.0, 2.5, 5.0, 10, and 20ug/ml. All Method 8270C QC criteria apply to the SIM analysis.

13.0 SAFETY

13.1 Always wear safety glasses or a shield for eye protection, gloves and lab coat.

13.2 Observe proper mixing when working with reagents and chemicals. Preparation of samples and standards should be handled under the hood.

13.3 A reference file of material data handling sheet is available to all personnel involving in these analyses.

14.0 POLLUTION PREVENTION

14.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

15.0 WASTE MANAGEMENT

15.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

16.0 DEFINITIONS

16.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

17.0 REFERENCES

- Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW846, 3rd Edition, Revision 3, December 1996

- DOD Quality System Manual, Final Version 3, March 2005

TABLE 1

Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

Semivolatiles	CAS Number	Quantitation Limits** Water ug/L	Low Soil/Sediment[b] ug/Kg
Benzaldehyde	100-52-7	10	330
Phenol	108-95-2	10	330
bis (2-Chloroethyl) ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
2-Methylphenol	95-48-7	10	330
bis (2-Chloroisopropyl) ether	108-60-1	10	330
Acetophenone	98-86-2	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
bis (2-Chloroethoxy) methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline***	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
Caprolactam	105-60-2	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene***	77-47-4	10	330
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	20	660
1,1'-Biphenyl	92-52-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline***	88-74-4	10	330

TABLE 1 (Cont)

Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

Semivolatiles	CAS Number	Quantitation Limits** Water ug/L	Low Soil/Sediment[b] ug/Kg
Dimethylphthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
2,6-Dinitrotoluene	606-20-2	10	330
3-Nitroaniline***	99-09-2	10	330
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol***	51-28-5	20	660
4-Nitrophenol***	100-02-7	20	660
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenyl-phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline***	100-01-6	10	330
4,6-Dinitro-2-methylphenol***	534-52-1	20	660
N-nitrosodiphenylamine	86-30-6	10	330
4-Bromophenyl-phenylether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Atrazine***	1912-24-9	10	330
Pentachlorophenol***	87-86-5	20	660
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butylphthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Pyrene	129-00-0	10	330
Butylbenzylphthalate	85-68-7	10	330
3,3'-Dichlorobenzidine***	91-94-1	20	660
Benzo(a)anthracene	56-55-3	10	330
Chrysene	218-01-9	10	330
bis (2-Ethylhexyl) phthalate	117-81-7	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330

TABLE 1 (Cont)

Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno (1,2,3-cd) pyrene	193-39-5	10	330
Dibenz (a,h) anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330

[b] Medium soil/sediment contract required quantitation limits (CRQL) for semivolatile TCL compounds are 60 times the individual low soil/sediment CRQL.

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment. Calculated on dry weight basis as required by the contract, will be higher.

*** Poor performers/sporadic marginal failures. Poor extraction efficiency, tendency to decompose, or poor chromatographic behavior.

TABLE 2

Appendix IX Compounds

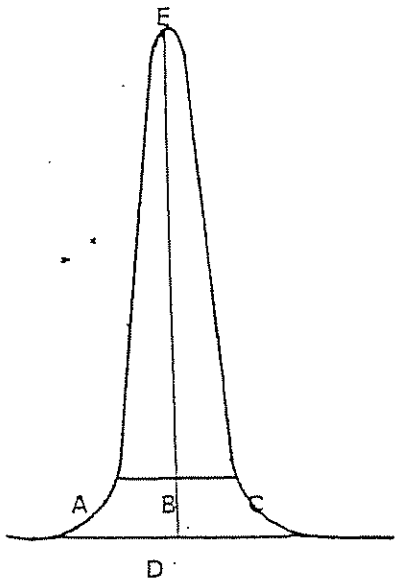
1. 2-Acetylaminofluorene
2. 4-Aminobiphenyl
3. Aniline
4. Chlorobenzilate
5. m-Cresol
6. Dimethoate
7. 4-Dimethylaminoazobenzene
8. 7,12-Dimethylbenz(a)anthracene
9. 3,3'-Dimethylbenzidine
10. a,a-Dimethylphenethylamine
11. 1,3-Dinitrobenzene
12. Diphenylamine
13. 1,4-Dioxane
14. Famphur
15. Hexachloropropene
16. Isodrin
17. Isosafrole
18. Kepone
19. Methapyrilene hydrochloride
20. 3-Methylcholanthrene
21. Methyl methane sulfonate
22. 1,4-Naphthoquinone
23. 1-Naphthylamine
24. 2-Naphthylamine
25. 4-Nitroquinoline-1-oxide
26. N-Nitrosodi-n-butylamine
27. N-Nitrosodiethylamine
28. N-Nitrosodimethylamine
29. N-Nitrosomethylethylamine
30. N-Nitrosomorpholine
31. N-Nitrosopiperidine
32. N-Nitrosopyrrolidine
33. 5-Nitro-o-toluidine
34. Pentachlorobenzene
35. Pentachloronitrobenzene
36. Phenacetin
37. 1,4-Phenylenediamine
38. Phorate
39. 2-Picoline
40. Pronamide
41. Pyridine
42. Safrole
43. 1,2,4,5-Tetrachlorobenzene
44. 2,3,4,6-Tetrachlorophenol
45. Thionazin
46. o-Toluidine
47. o,o,o-Triethylphosphorothioate
48. sym-Trinitrobenzene
49. Methyl methacrylate
50. Ethyl methacrylate
51. Ethyl methane sulfonate
52. o-Anisidine
53. p-Cresidine
54. 5-Chloro-2-methylaniline
55. Phthalic anlydride
56. 2,4-Diaminotoluene
57. 1-Chloronaphthalene
58. Diallate
59. Disulfoton
60. Methylparathion
61. Ethylparathion
62. 4-Aminoazobenzene
63. 3,3-Dimethoxybenzidine
64. Tetraethyldithiopyrophosphate
65. 2,6-Dichlorophenol
66. Aramite
67. Hexachlorophene

TABLE 3

ADDITIONAL COMPOUNDS

1. Chloropicrin
2. Malononitrile
3. Thiodiglycol
4. 1,4-Oxathiane
5. 1,4-Dithiane
6. Diisopropyl Methylphosphonic Acid
7. Dimethyl Methylphosphonic Acid
8. 2-Chlorobenzilidenemalononitrile
9. 2-Chloroacetophenone
10. 3-Chloroacetophenone
11. 4-Chloroacetophenone
12. 2-Chlorobenzaldehyde
13. 3-Chlorobenzaldehyde
14. 4-Chlorobenzaldehyde
15. 2-Hydroxyacetophenone
16. 3-Hydroxyacetophenone
17. 4-Hydroxyacetophenone
18. Benzyl Alcohol
19. Benzoic acid***
20. 1,2-Dichlorobenzene
21. 1,3-Dichlorobenzene
22. 1,4-Dichlorobenzene
23. 1,2,4-Trichlorobenzene

Figure 1
Peak Tailing Factors



Peak Tailing Factor = BC/AB

Sample calculation: Peak Height = DE = 100mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23mm
AB = 11 mm
BC = 12 mm
Tailing Factor = 12/11 = 1.1

SOP No: N.1

Title: Flash Point

Scope: This method describes the procedure for determining the flash point of liquids including those that tend to form a surface film under test conditions. Liquids containing non-filterable, suspended solids are also tested using this method. This method is also applicable for soils, solids and waste matrices.

1.0 SUMMARY OF METHOD

1.1 The sample is placed in the cup of the Pensky-Martens tester up to the engraved line and tested for flash in 2-3 degree increments starting at room temperature until it flashes or up to 100°C. The flash point is the lowest temperature at which the application of the test flame ignites the vapor above the sample.

2.0 REFERENCES

- Method 1010: Pensky-Martens closed-cup method for determining ignitability (Revision 0; September 1986).

3.0 EQUIPMENT AND SUPPLIES

- Pensky-Martens closed-cup tester
- Thermometer, calibrated (range: 0 to 100°C)
- Spatula
- Propane gas cylinder
- Lighter
- Aluminum weighing dish
- Fume hood

4.0 REAGENTS

- p-Xylene (Quality Control Standard)

5.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

5.1 Samples are collected in appropriately sized glass bottles, which have been precleaned and certified to be free of target analytes. Samples are shipped in coolers packed with ice and stored at the lab in refrigerators at 2-6°C until analysis.

6.0 PROCEDURE

6.1 Liquid

6.1.1 Verify the flammability of the sample by placing a few drops of the sample on the aluminum weighing dish and then place a lighted splinter about ¼" above the sample. If sample is flammable, report as R (flammable at room temperature). Do not perform the remaining steps.

If the sample is not flammable, proceed to step 6.1.2.

6.1.2 Pour sample (approximately 50mls) to pre-measured marking engraved into the cup of the Pensky-Martens apparatus.

6.1.3 Insert the cup into the heating element of the apparatus.

6.1.4 Place the cup-closure containing the thermometer on to the cup and stir the waste using a variable speed motor and stirring rod.

6.1.5 Test for flash point in increments of 2-3°C up to 100°. If the sample flashes, record the temperature when it first flashed in the result column.

6.1.6 If the sample does not flash, heat until the sample reaches boiling point, or 100°C. Record temperature as N (temperature).

6.1.7 Allow the cup and thermometer to cool to room temperature before proceeding to the next sample.

6.1.8 Laboratory duplicate must be analyzed every 20 samples per matrix type.

6.2 Soil

6.2.1 Verify the flammability of the sample by placing a few grams of soil on the aluminum weighing dish and then place a lighted splinter about ¼" above the sample. If sample is flammable, report as R. Do not perform the remaining steps.

If the sample is not flammable, proceed to step 6.2.2.

6.2.2 Place sample up to the line engraved on cup of the Pensky-Martens apparatus.

6.2.3 Insert the cup into the heating element of the apparatus.

6.2.4 Place the cup-closure containing the thermometer on to the cup.

6.2.5 Test for flash point in increments of 2-3°C up to 100°. If the sample flashes, record the temperature when it first flashed in the results column.

6.2.6 If the sample does not flash, record as N(100).

6.2.7 Allow the cup to cool to room temperature before proceeding to the next sample.

6.2.8 Laboratory duplicate must be analyzed every 20 samples per matrix type.

6.3 Quality Control Standard (p-Xylene)

6.3.1 Treat p-Xylene as you would a liquid sample, starting from step 6.1.2. Record the flash point temperature for the standard. Analyze the p-Xylene at the end of each analytical batch, the acceptance criteria for both results must be from 26.4 to 28.0°C.

6.4 Quality Control (Samples)

6.4.1 Perform a duplicate analysis for each matrix per batch.

6.5 Safety

6.5.1 The nature of this analysis has a potentially high risk of a fire break-out, therefore, it is essential that a fire extinguisher be available for such as emergency. The class of fire extinguisher to be used is "B or C".

6.5.2 The Pensky-Martens tester must be performed in a fume hood.

6.5.3 Lab coat, face shield and insulated gloves should be worn while performing the test.

7.0 POLLUTION PREVENTION

7.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

8.0 WASTE MANAGEMENT

8.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

9.0 DEFINITIONS

9.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

SOP No: M.5

Title: Volatile Organics - Method 8260B

1.0 SCOPE AND APPLICATION

1.1 This method is used to identify and quantify purgeable compounds in waste, water, soil, and various types of waste samples using GCMS methodology. The compounds listed below include the most common requested volatile parameters.

Target Compound List

Dichlorodifloromethane
Chloromethane
Vinyl Chloride
Bromomethane
Chloroethane
Trichlorofluoromethane
1,1-Dichloroethene
1,1,2-Trichloro-1,2,2-Trifluoroethane
Acetone
Carbon Disulfide
Methyl Acetate
Methylene Chloride
Trans-1,2-Dichloroethene
Methyl tert-Butyl Ether
1,1-Dichloroethane
cis-1,2-Dichloroethene
2-Butanone
Chloroform
1,1,1-Trichloroethane
Cyclohexane
Carbon Tetrachloride
Benzene
1,2-Dichloroethane
Trichloroethene
Methylcyclohexane
1,2-Dichloropropane

Bromodichloromethane
Cis-1,3-Dichloropropene
4-Methyl-2-Pentanone
Toluene
Trans-1,3-Dichloropropene
1,1,2-Trichloroethane
Tetrachloroethene
2-Hexanone
Dibromochloromethane
1,2-Dibromoethane
Chlorobenzene
Ethylbenzene
Xylenes (total)
Styrene
Bromoform
Isopropylbenzene
1,1,2,2-Tetrachloroethane
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
1,2-Dibromo-3-chloropropane
1,2,4-Trichlorobenzene

Appendix IX List

** Acetonitrile
** Acrylonitrile*
Allyl Chloride
** 2-Chloro-1,3-butadiene
Dibromomethane
trans-1,4-Dichloro-2-butene
p-Dioxane*
Ethyl methacrylate
Isobutyl alcohol
Methacrylonitrile
Methyl iodide
Methyl methacrylate
Pentachloroethane
Propionitrile*
1,1,1,2-Tetrachloroethane
Hexachloroethane
1,2,3-Trichloropropane
1,1-Dichloropropylene
1,4-Dioxane
** Acrolein
Bromochloromethane

* Poor purging efficiency /**poor performers resulting in high EDL.

Additional Compounds

2, 2 - Dichloropropane
1, 3 - Dichloropropane
Bromobenzene
n - Propylbenzene
2 - Chlorotoluene
4 - Chlorotoluene
1, 3, 5 - Trimethylbenzene
tert - Butylbenzene
1, 2, 4 - Trimethylbenzene
sec - Butylbenzene
4 - Isopropyltoluene
n - Butylbenzene
Hexachlorobutadiene
Naphthalene
1, 2, 3 - Trichlorobenzene
Iodomethane

TABLE 1

Comp No.	Compound Name	Quantitation Ion
1)	Dichlorodifluoromethane	85
2)	Chloromethane	50
3)	Vinyl Chloride	62
4)	Bromomethane	94
5)	Chloroethane	64
6)	Trichlorofluoromethane	101
7)	1,1-Dichloroethene	96
8)	1,1,2-Trichloro-1,2,2-Trifluoroethane	101
9)	Acetone	43
10)	Carbon Disulfide	76
11)	Methyl Acetate	43
12)	Methylene chloride	84
13)	trans-1,2-Dichloroethene	96
14)	Methyl tert-Butyl Ether	73
15)	1,1-Dichloroethane	63
16)	Cis-1,2-Dichloroethene	96
17)	2-Butanone	43
18)	Chloroform	83
19)	1,1,1-Trichloroethane	97
20)	Cyclohexane	56
21)	Carbon Tetrachloride	117
22)	Benzene	78
23)	1,2-Dichloroethane	62
24)	Trichloroethene	130
25)	Methylcyclohexane	83
26)	1,2-Dichloropropane	63
27)	Bromodichloromethane	83
28)	cis-1,3-Dichloropropene	75
29)	4-Methyl-2-pentanone	43
30)	Toluene	91
31)	trans-1,3-Dichloropropene	75
32)	1,1,2-Trichloroethane	97
33)	Tetrachloroethene	164
34)	2-Hexanone	43
35)	Dibromochloromethane	129
36)	1,2-Dibromoethane	107
37)	Chlorobenzene	112
38)	Ethylbenzene	106
39)	Xylene (total)	106
40)	Styrene	104
41)	Bromoform	173
42)	Isopropylbenzene	105
43)	1,1,2,2-Tetrachloroethane	83

44)	1,3-Dichlorobenzene	146
45)	1,4-Dichlorobenzene	146
46)	1,2-Dichlorobenzene	146
47)	1,2-Dibromo-3-Chloropropane	75
48)	1,2,4-Trichlorobenzene	180

*Poor performers.

2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into a gas chromatograph by the purge and trap method.

2.2 An inert gas is bubbled through the solution at ambient temperature (at elevated temperatures for soil samples), and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is heated to elute the components, which are detected with a mass spectrometer.

3.0 INTERFERENCES

3.1 Interferences purged from the samples will vary considerably from source to source, depending upon the particular sample or extract being tested. The analytical system, however, should be checked to ensure freedom from interferences and contamination. All samples and QC samples must be examined for the possibilities of contamination or carryover. If high concentrations of analyte(s) are found in a sample, the next sample(s) on the sequence batch should be checked for possible carryover. Concentrations comparable to the highest levels of the calibration range are considered a potential source of carryover. Usually those compounds that are quantified against the second and third internal standards (applicable to volatiles) may cause carryover. After the analysis of a sample containing high concentration of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the organic compounds present in the high level sample, freedom from contamination has been established. If the sample is suspected to have carryover, re-analysis should be performed. The ALS (Automatic Liquid Sampler) position that contained the sample with the high levels of contaminants needs to be marked and cleaned. The next sample loaded in that position needs to be monitored for any possibilities of carryover. The contaminated position and corrective action performed must be documented on the injection log. Contamination generated by non-target compounds also needs to be monitored. The level of carryover by non-target compounds should be compared to the internal standards. The significance of the non-target

compound carryover should be evaluated by the supervisor to determine the overall impact on the sample results.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph

Hewlett-Packard 5890 Gas Chromatographic Systems (GC) are employed for execution of this method. The GCs are complete with temperature programming capabilities, and all required accessories such as columns, gases, cooling valves and syringes. Sample introduction is accomplished through the use of purge and trap systems.

4.2 Purge and Trap System

The Tekmar 2000/3000 purge and trap system is coupled with the gas chromatographic system. The system is equipped with a Tekmar 2016 sixteen position autosampler, and a sample heating device for soil samples.

The purge and trap device consists of three separate modules. The sample purger, the trap and the desorber. The purging chamber accepts glass vessels capable of holding at least 5-ml of liquid samples or 5 grams of soil samples. The glass tubes are stored in an oven at 105°C to prevent contamination from organic vapors.

The trap utilized by the laboratory is 25 cm long and has an inside diameter of 0.105 in. Traps are marketed as VOCARB 3000 by vendors such as Supelco, and consist of graphitized carbon.

A complete description of the inner workings of this purge and trap model is available in the Tekmar 2000/3000 manual.

4.3 Mass Spectrometer

The HP-5970 quadruple Mass Selective Detector is used as the detection device for Method 8260B. This system is capable of scanning from 35-260amu every one second or less, using 70 volts (nominal) electron energy in the EI mode. After proper tuning, the system produces a mass spectrum that meets all the criteria in Table 4 when 50ng of 4-Bromofluorobenzene (BFB) are injected into the gas chromatograph.

4.4 Data System

Each GC/MS system is attached to a dedicated 486 or pentium based computer equipped with Enviroquant software for automated acquisition and processing. The software generates total and extracted ion profiles of each compound and is capable of performing library searches on spectra using a full EPA/NIH Mass

Spectral Data Library. Each system is attached to an internal laboratory computer network for additional data processing, storage and archiving.

4.5 Column

Megabore column, 60 meters in length, with a 0.53 mm internal diameter and 2.0 micron film thickness is used.

4.6 Microsyringes - 10ul, 25ul, 100ul, 250ul, 500ul, and 1000ul.

4.7 Luerlock syringe - 5 and 25ml gas tight.

5.0 REAGENTS AND STANDARDS

5.1 Organic free reagent water.

5.2 Stock solutions.

5.3 Methanol, High purity grade, B & J Brand for Purge and Trap analysis.

6.0 SAMPLE COLLECTION AND PRESERVATION

6.1 Water samples may be collected in glass containers having a total volume of at least 40mL with a teflon-lined septum and an open top screw-cap. Soil samples may be collected in glass containers or closed end tubes (e.g., brass sleeves) in sufficient quantity to perform the analysis. Headspace should be avoided. The specific requirements for site sample collection are outlined by the Region.

6.2 For collection of water samples, the containers must be filled in such a manner that no air bubbles pass through the sample as the container is being filled. Seal the vial so that no air bubbles are entrapped in it.

6.3 Water samples are preserved to a pH of 2 at the time of collection.

6.4 All samples must be iced or refrigerated at 4°C (±2°C) from the time of collection until analysis.

6.5 All water and soil samples must be analyzed within 14 days of collection.

7.0 METHOD DETECTION LIMIT

7.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit official book.

8.0 METHOD PERFORMANCE

8.1 The MDL concentrations listed in the GPL MDL book are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and a reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Calibration

Five concentration levels are analyzed for each analyte and surrogate standards. A minimum five point curve is then created after all 5 points are analyzed. Calibration levels are analyzed at 5, 20, 50, 100, 150 and/or 200ug/l for all analytes and surrogate standards except for 2-Chloroethyl vinyl ether which is analyzed at 40, 100, 200, 300 and 400ug/l.

9.2 Instrumentation Parameters

The following set of operating conditions exist for method 8260B.

Electron energy:	70 volts (nominal)
Mass range:	35-260amu
Scan time:	Not to exceed 1 second/scan
Initial column temperature:	10° C
Initial column holding time:	4 min
Column temperature program:	8° C/minutes
Final column temperature:	190° C
Final column holding time:	1 min
Injector temperature:	220° C
Source temperature:	According to manufacturer's specifications
Transfer line temperature:	280° C (Preset to manufacturer)
Carrier gas:	Helium at about 8ml/min

9.3 Tuning

9.3.1 At the beginning of each day, each GC/MS system is injected with 50ng of BFB and tuned to meet the criteria listed in Table 4. The analysis must not commence unless the criteria are met. This requirement must be met for each 12 hour interval.

9.3.2 One of the following approaches is used to evaluate the BFB tune.

- One scan at the apex without background subtraction.
- Three scans (the apex peak scan and the scan immediately preceding and following the apex) are acquired and averaged.
- Use the mean of the apex and the preceding of the following scans.
- Use the average across the entire peak.

9.4 Calibration Requirements

Once tuning requirements are met, the initial or continuing calibration check must be established. The generation of response factors is the next step in establishing the calibration requirements for method 8260B. The instructions for determining the response factors are as follows:

9.4.1 Tabulate the area response of the quantitation ions (see Table 1) against the concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard with the retention time closest to the compound being measured. The RF is calculated as follows:

$$RF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

where:

A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard.
C_x = Concentration of the compound being measured.

The average RF must be calculated for each compound. A system performance check should be completed before the calibration curve is used. Five compounds, the System Performance Check Compounds, or SPCCs, are checked for a minimum average response factor. These compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene (see Table 3). The minimum relative response factors for these compounds should be 0.1, 0.1, 0.1, 0.3 and 0.3. These compounds typically have RFs respectively, of 0.4-

0.6 and are used to check compound instability and check for degradation caused by contaminated lines or active sites in the system. Initial calibration is analyzed at a minimum of 5 levels. These levels are 5, 20, 50, 100, 150 and/or 200ug/l. Acrolein and acrylonitrile have higher initial calibration levels.

	Levels					
	5ppb	20ppb	50ppb	100ppb	150ppb	200ppb
Acrolein	25	100	250	500	750	1000
Acrylonitrile	25	100	250	500	750	1000
2 - Chloroethyl Vinyl Ether	20	80	200	400	600	800

Using the RFs from the initial calibration, calculate the percent relative standard deviation (% RSD) for all compounds.

$$\%\ RSD = \frac{SD \times 100}{x}$$

where:

RSD = relative standard deviation
X = mean of 5 initial RFs for a compound
N = Number of calibration points
SD = standard deviation of average RFs for a compound
X_i = Response factor for each point

$$SD = \frac{\sum_{i=1}^{N} (Xi-X)}{N-1}$$

9.4.2 After analyzing the initial calibration standard, average response factor RF should be calculated for each compound. The percent relative standard deviation %RSD should be equal or less than 15% for each compound. However, Calibration Check Compounds (CCC's) must have a %RSD less than or equal to 30%. It is likely that some analytes may exceed the 15% acceptance limit for the %RSD. In those instances the following steps should be considered:

- If the %RSD is greater than 15%, the analyst should review the results (area counts, response factors, standard concentrations) for those analytes to ensure that the problem is not associated with a single standard. If so, that standard should be reanalyzed and RSD recalculated. Replacing of standard may be necessary in some cases.

- It may also be necessary to narrow the calibration ranges to achieve a better linearity. High standard may saturate the column or/and the detector and need to be dropped from the calibration curve accordingly. Similarly poor purging compounds that exhibited erratic chromatographic behavior in the lowest calibration point could also be reviewed and dropped if necessary. The changes to the upper end of the calibration will affect the need to dilute samples above that range, while the change to the lower calibration will affect the sensitivity and elevate the reporting limit of the method. When considering dropping one or two points to narrow the calibration range, a minimum of five points is required for curve to be acceptable.

9.4.2.1 After visual inspection of the calibration points for each analytes and for those analytes that did not meet the %RSD criteria, at the discretion of the analyst it should consider to employ a regression equation that does not through the origin or a quadratic (second order) model. Linearity is presumed acceptable if the correlation coefficient (r) is equal to or greater than 0.995.

9.4.2.2 If linear regression curve did not match the response being observed, quadratic equation should be considered as an option. A quadratic model requires six standard. Linearity is presumed acceptable if the correlation coefficient (r^2) is equal to or greater than 0.99.

9.4.2.3 Prior to use for sample analysis, the acceptability of initial calibration curve must be verified through analysis of calibration verification (ICV) solution obtained from second source. Calibration verification analysis should meet the same acceptance criteria used for continuing (daily) calibration.

9.4.3 Continuing (Daily) Calibration

The initial calibration curve for each compound of interest must be checked and verified once every 12 hours of analysis time. This is accomplished by analyzing a 50ppb calibration standard and verifying if all compound of interest meet the acceptance criteria for daily calibration. Daily calibration standard analysis must meet the relative response factor criteria for SPCC and non SPCC % difference criteria of calibration check compounds (CCC) and non CCC. If SPCC and non SPCC criteria listed in Table 3 are not met, the system must be evaluated and corrective action must be taken before sample analyses begins. Potential problem include standard mixture degradation, injection port inlet contamination, contamination of front end of the analytical column, and active sites in the column or chromotographic system. After the criteria for relative response factor (RRF) are met, calculate the % difference of each compound to check the validity of the initial calibration.

Calculate the % difference using the following equation:

$$\%\text{Difference} = \frac{RRF_c - RRF_i}{RRF_i} \times 100$$

where:

RRF_c = Relative response factor from continuing calibration standard

RRF_i = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

If the 20 percent difference for each CCC and non-CCC (except 8 poor purging or performing compounds that could have %D less than or equal to 40%) did not meet, corrective action should be taken. After corrective action, if the source of the problem cannot be determined, a new five point calibration MUST be generated. This criteria must be met before sample analysis begins. The internal standard response and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. Tables 2 and 3 list SPCC and CCC compounds.

9.4.4 When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of manual integration and notation of date and initials of person performing the task.

9.5 Retention Time and Area Change

9.5.1 If the retention time of any internal standard of a sample or QC sample change by more than 30 seconds from the daily calibration verification standard or if the daily calibration verification standard change by more than 30 seconds from mid level standard of the latest initial calibration corrections must be made. Affected samples must be reanalyzed after problem is corrected.

9.5.2 If internal standard area in the samples changes by a factor two (-50% to +100%) from the calibration standard verification, corrections must be made. After corrections reanalysis of samples analyzed while the system was malfunctioning is required. Area of the daily calibration standard verification must not change by a factor of two (-50% to +100%) from that in the midpoint standard of the most recent initial calibration.

10.0 DATA ANALYSIS AND CALCULATION

10.1 Qualitative Analysis

10.1.1 The compounds listed in Section 1 shall be identified by an analyst by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identification.

10.1.1.1 Relative relation time of the sample and standard must agree ±0.05.

10.1.1.2 Correspondence of the sample component and standard component mass spectra.

10.1.2 The requirement for qualitative verification by comparison of mass spectra are as follows:

All ions present in the standard mass spectra at a relative intensity greater than 10% must be present in the sample spectrum. All ions specified must agree within ±20% between the standard and sample spectra. Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for.

10.2 Quantitative Analysis

When a compound is identified, quantification is based on the integrated abundance of the primary characteristics ion and the response and amount of the corresponding internal standard.

$$\text{Concentration (ug/L)} = \frac{(Ax)(Iis)(D)}{(Ais)(Avg\ RF)(Vo)}$$

$$\text{Concentration (ug/kg)} = \frac{(Ax)(Is)(D)}{(Ais)\ (Avg\ RF)\ (W)\ (S)}$$

where:

Ax	=	Area of primary ion
Is	=	Amount of internal standard, ug/L
Ais	=	Area of ion for internal standard
Avg RF	=	Mean relative response factor for compound measured
Vo	=	Volume of sample purged, L
D	=	Dilution factor
W	=	Weight in Kg
S	=	% Solid

10.3 For non-TCL components, a library search may be executed. Up to 20 non-target organic compounds shall be tentatively identified when required. Guidelines for making tentative identification:

10.3.1 Ions greater than 10% of the most abundant ion should be present in the sample spectrum.

10.3.2 Relative intensities of the major ions should agree within ±20%.

10.3.3 Molecular ion present in the reference spectrum should be present in sample spectrum.

.1.1 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.0 PROCEDURE

11.1 Sample Preparation

11.1.1 For sample preparation and introduction of close system purge and trap and extraction of soil and water volatile compounds to the mass spectrometer, refer to GPL SOP M.7.

11.2 Water Samples

11.2.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

11.2.2 BFB tuning criteria and daily GC/MS calibration criteria must be met before analyzing samples.

11.2.3 Adjust the purge gas (Helium) flow rate to approximately 40ml/min on the purge and trap device.

11.2.4 Remove the plunger from a 5ml syringe. Open the sample and carefully pour the sample into the syringe barrel to just short of overflowing. Compress the sample. Vent any residual air while adjusting the sample volume to 5.0ml.

11.2.5 Add 5.0ul of surrogate spiking solution and 5ul of internal standard solution (50ppm each) through the valve bore of the syringe and load samples into the sample tube. Immediately after loading the sample, verify the pH of the samples using narrow range pH paper. Sample vials should not be opened to verify pH prior to loading into the purge unit.

11.2.6 Lab Control Spikes (LCS) is analyzed per batch or every 20 samples and at the same frequency as the method blank. LCS is spiked with all target

compound of interest. LCS compound must meet the criteria outlined in the Quality Control section 12.7.

11.2.7 For the matrix spike analysis, add 5ul of the matrix spike solution (50ppm) to the sample to be purged. This will yield a final concentration of 50ug/L (ug/kg for soils) in the final sample. Matrix spike QC sample must contain all target analytes of interest.

11.2.8 The samples are analyzed with appropriate dilution when the concentration level of any analyte exceeds the calibration range. Dilutions are made in the 5ml gas tight luer lock syringe by adding reagent water. Calculate the volume of reagent water needed for the dilution and add into a 5ml syringe. Dilution of samples should result in analysis with the highest concentration target analyte in the upper half of the calibration range. Using a suitable syringe, add the exact volume of sample into the reagent water in the 5ml syringe. Add 5ul of Internal Standard & Surrogate and analyze as discussed above. If the dilute sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution.

11.2.9 The spiked sample is injected into a purging tube attached to the Purge & Trap device. Purge the sample for 11 minutes and desorb the contents of the trap at 200° for 2 minutes into the GC/MS.

11.2.10 Low Concentration Method: This method is applicable to water samples containing low levels of contaminants. The detection limits are generally improved by analyzing larger sample volume (25ml). The detection limits of compounds with poor purging efficiency (such as ketones) do not significantly improve when low concentration method is utilized. The low concentration analytical procedure is virtually the same as low level water analysis except for concentration of the calibration, surrogate and internal standards. The following concentration levels and ranges are analyzed:

Calibration	-	.5, 1, 10, 20, 40, 60ug/L
Surrogates	-	.5, 1, 10, 20, 40, 60ug/L
Internal Standards	-	10ug/L
Continuing Calibration	-	10ug/L

Acceptance criteria for the initial and continuing calibrations is the same as the normal –5mL purge. Criteria for percent RSD and difference in the initial and continuing calibrations are described in section 8.4. Minimum response factor for the poor purging or poor performers is 0.010.

11.2.11 Cleaning of purge and trap cells.

Upon completion of volatile analysis, the purge and trap cells are disconnected from the ALS. The purge and trap cells are cleaned with deionized water and mild soap, and rinsed with deionized water. The purge and trap cells are then baked in an oven at 105° for at least four hours. Upon completion of baking, the purge and trap cells are placed in a container covered with aluminum foil.

11.3 Sediment/Soil and Waste Samples

11.3.1 Low Concentration method: Applicable for samples containing individual compounds of <1mg/kg. All granular/porous waste/sediment/soil samples can be analyzed using this method.

Weigh 5g of the sample into a glass sparge tube and record the weight to the nearest 0.1g. Add reagent water to a 5ml luerlock type syringe and adjust the volume to 5ml. Add 5ul of internal standard and 5ul of surrogate standard to the water. Connect the sparge tube with the sample to the purge & trap device and add the spiked water to the sample. Heat the sample to 40°C $\pm$1°C and purge the sample for 11 minutes. Desorb the trap at 200°c for 2 minutes into the GCMS.
If the concentration level of any analyte exceeds the calibration range and is below 1mg/kg, the sample should be analyzed by weighing 1g of the sample. If the concentration level of any analyte is higher than 1mg/kg, then the high concentration method should be used.

11.3.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

11.3.3 BFB tuning criteria and daily GC/MS calibration criteria must be met before analyzing samples.

11.3.4 Adjust the purge gas (Helium) flow rate to approximately 40ml/min on the purge and trap device.

11.3.5 Weigh 5.0 grams of the soil sample into a tared purge device. Use a top loading balance. Note and record the actual weight to the nearest 0.10g. Quickly connect the device to the purge and trap system.

11.3.6 Prepare a soil method blank which consists of a 5g of a purified solid matrix to reagent water with 5ul of surrogate and 5.0ul of internal standard solution (50ppm each). Add the solution through the valve pore of the syringe into the soil sample tube.

11.3.7 Lab Control Spikes (LCS) is analyzed per batch or every 20 soil/sediment samples and at the same frequency as the method blank. LCS is spiked with all target compound of interesting in a purified solid matrix. LCS

compound must meet criteria specified in the Quality Control section 12.7.

11.3.8 For the matrix spike analysis, add 5ul of the matrix spike solution (50ppm) to the sample to be purged. This will yield a final concentration of 50ug/L (ug/kg for soils) in the final sample. Matrix spike QC sample must contain all target analytes of interest.

11.3.9 High Concentration Method: The method is based on extracting the soil/sediment with methanol. Weigh 4gm of the sample into a 20ml vial using a top loading balance. Record the weight to the nearest 0.1g. Using a pipet, add 9.0ml methanol; then add 1.0ml of the surrogate spiking solution into the vial. Close the cap and shake for 2 minutes. Pipet approximately 1ml of the extract to a GC vial for storage, using a disposable pipet. Transfer approximately 1ml of the method blank to a separate GC vial for each set of samples. The standards, LCS and blanks should contain 100µl of a solvent to simulate the sample condition. 100ul of the extract is added to reagent water collected in a 5ml luerlock syringe. 5ul each of Internal Standard is added to the syringe. Proceed with the analysis as outlined in the water sample analysis. If further dilution is needed, a smaller extract volume is used and the same procedure is repeated.

A separate method blank containing 100ul methanol should be analyzed prior to a high concentration sample.

11.3.10 For matrix spike in the high concentration sediment/soil samples, 8.0ml of methanol, 1.0ml of surrogate spike solution, and 1.0ml of spike solution is added to a 4g sample. Cap and shake for 2 minutes. Add a 100µl aliquot of this extract to 5ml of organic free reagent water for purging.

11.3.11 Samples extracted through Toxicity Characteristic Leachate Procedure by zero headspace extractor are analyzed by this method. Only those compounds designated by the TCLP method are accounted for. Samples are analyzed at 10 times dilution in order to minimize the effects of acetic acid on the chromatography and purge and trap systems.

12.0 QUALITY CONTROL

12.1 Prior to initiating on going data, it is necessary that the GC/MS meets BFB abundance criteria (Table 4) every twelve (12) hour period for the method.

12.2 When twelve (12) hours have elapsed since the initial tune, the GC/MS must be returned to continue the analysis.

12.3 Internal standard responses and retention times in the calibration checks, blanks and samples must be evaluated. Retention time variation of any internal standard in the calibration standard must be less than 30 seconds from that in the midpoint standard in the calibration standard. All other samples and QC samples must be compared to the daily CCV and should not vary by more than or equal to 30 seconds. If this criteria exceeds samples that did not meet the criteria should be re-injected.

12.4 Surrogate recoveries must be evaluated by determining whether the concentrations fall within internal QC limits. If surrogate recoveries falls outside the established QC limits, then analyze the sample that fail the criteria.

12.5 A matrix spike and spike duplicate must be performed every tune or 20 samples, whichever comes first, at the concentration equal to mid-level calibration (50ug/l). If matrix spike QC recoveries does not meet established acceptance criteria, the analyst and the manager must assess the batch to determine whether the spike results are attributable to matrix affect, or the result of other problems in analytical process. If all batch QC elements which are not affected by matrix are in control (e.g., method blank, LCS) the poor recovery may be attributed to matrix affect. If the native concentration is low, and the MS/MSD recoveries confirm matrix interferences, dilute the MS/MSD and reanalyze. TCLP samples are spiked with TCLP target compounds at 50ug/l concentration.

12.6 A method blank must be performed for each 12 hour time period.

12.6.1 A method blank for volatile analysis must contain less than or equal to ½ of the reporting limits. Any method blank must comply with the following criteria:

- If the concentration of target analyte in the method blank is greater than MDL but less than the reporting limit, rinse the purging apparatus with two portions organic free reagent water and reanalyze the method blank. After corrective action, if low level contamination is still detected, associated sample data may be reported with qualifier.

- If the concentration of any target analyte in the method blank is greater than the reporting limit, it may be necessary to wash the purging device with a soap solution, rinse it with organic free reagent water, and then dry the purging device in an oven at 105°C. No sample should be analyzed before the contamination is eliminated.

12.7 Lab Control Spikes (LCS) is analyzed per batch or every 20 samples and at the same frequency as the method blank. LCS is spiked with all target compound of interest. Statistical control limit are based on 20 data points. Data points used in the data set must not be selectively included or excluded.

Each LCS must be evaluated against the Control Limits and Marginal Exceedance limit. If any LCS is outside the Control Limit, it should be also compared to the Laboratory Marginal Exceedance Limits to ensure that it does not exceed. If a single analyte in the LCS exceeded the Marginal Exceedance Limit, the LCS had failed and corrective action needs to be initiated.
If the LCS has more than the allowable number of Marginal Exceedance, the LCS has failed and corrective action needs to be initiated.

The corrective action for failed LCS is based on professional judgment in conjunction with matrix spike and surrogate recoveries in the same batch. If after checking the associated QC's it was determined by the section supervisor or manager that the LCS had failed, all affected samples associated with the out of control LCS should be reprocessed and re-analyzed.

If samples cannot be reprocessed due to lack of sample volume or the holding times has lapsed, the results should be reported with appropriate flag. The report case narrative must include a discussion of the failed LCS, and its impact on the data quality.

When LCS is spiked with large number of analytes, the laboratory should add up total number of exceedances for the LCS based on the number of analyte spiked in the LCS. The total of exceedance should be compare with the allowable number from the following chart.

Since a large amount of GPL's clients most requested compound list are fairly long (> 20 compounds), the laboratory will spike all the reportable compounds as specified in the clients project. At a minimum, 16 compounds from the list will be selected and used to verify if the LCS meets the required QC requirements. However, the laboratory should ensure that all compounds in the target list are used to verify the LCS criteria over a period of two years.

Number of analyte in the LCS	Allowable Number of Marginal Exceedances of LCS
< 11	0
11-30	1
31-50	2
51-70	3
71-90	4
>90	5

Since a large amount of GPL's clients most requested compound list are fairly long (> 20 compounds), the laboratory will spike all the reportable compounds as specified in the clients project. At a minimum, 16 compounds from the list will be selected and used to verify if the LCS meets the required QC requirements. However, the laboratory shall ensure that all compounds in the target list are used to verify the LCS criteria over a period of two years.

12.8 QC limits for MS/MSD, LCS and surrogates are established semi-annually for internal use.

12.9 For DOD projects, DOD QSM control limits policy shall be followed for MS/MSD, LCS and surrogates.

13.0 MS SIM ANALYSIS

13.1 When requested, low concentration target analytes can be recovered using Selective Ion Monitoring (SIM) mode. Selective Ion Monitoring (SIM) mode is a data acquisition technique in which only a few selected ion fragments are monitored in order to obtain maximum selectivity. Standard spike volumes for surrogates, internal standards, matrix spikes, and laboratory control samples are dependent on the reporting limit requested.

13.2 Five calibration standard levels are analyzed at concentrations dependent on the reporting limit requested. All Method 8260B QC criteria applies to the 8260 SIM analysis.

14.0 SAFETY

14.1 Safety glasses, laboratory coats, and latex gloves must be worn.

14.2 Due to the toxicity or carcinogenicity of each reagent, each chemical compound should be treated as a potential health hazard.

14.3 Material Safety Data Sheets (MSDS) can be found on the procurement bookshelves located in the administrative area.

14.4 Standard preparation should be handled under a hood.

15.0 POLLUTION PREVENTION

15.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

16.0 WASTE MANAGEMENT

16.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

17.0 DEFINITIONS

17.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

18.0 REFERENCES

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Revision 3, December 1996.

- DOD Quality System Manual, Final Version 3, January 2006

TABLE 2

CALIBRATION CHECK COMPOUNDS

1 - 1,1-Dichloroethene

2 - Chloroform

3 - 1,2-Dichloropropane

4 - Toluene

5 - Ethylbenzene

6 - Vinyl chloride

TABLE 3

SYSTEM PERFORMANCE CHECK COMPOUNDS

1 - Chloromethane

2 - 1,1-Dichloroethane

3 - Bromoform

4 - 1,1,2,2-Tetrachloroethane

5 - Chlorobenzene

All other compounds must meet a minimum RRF of 0.050

TABLE 4

BFB KEY ION ABUNDANCE CRITERIA

Mass	Ions Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

SOP No: J.43

Title: Cyanide, Total (colorimetric, manual spectrophotometric)

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure describes the manual colorimetric measurement of free (non-complexed) cyanide and hydrocyanic acid in water and soil samples after distillation for Total Cyanide in accordance with SW846 method 9014.

2.0 PURPOSE

2.1 This method details the procedure for the colorimetric measurement of free cyanide in water, wastewater, leachates, or distillates. The colorimetric method is suitable for concentrations between 5 and 200ug/L CN. Samples with higher levels can be diluted to fall within this range.

2.2 Total Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system under acidic conditions and in the presence of magnesium ion. The liberated HCN is absorbed in a scrubber containing NaOH solution. The cyanide in the scrubber solution is converted to cyanogen chloride by reaction with chloramine-T at a pH < 8. Color is formed on the addition of pyridine-barbituric acid. The color is measured at 578nm. The intensity of the color is proportional to the cyanide concentration.

3.0 REFERENCES

- SW846 method 9014.

4.0 SAMPLE HANDLING AND PRESERVATION

4.1 Refer to Method 9010B (GPL SOP J.3) for a discussion of potential cyanide interferences and how they can be reduced or eliminated.

4.2 Samples should be preserved with NaOH to a pH >12 when taken, and stored at 4°C. Analysis must be performed within 14 days of sampling. Distillates should be stored at 4°C in tightly sealed flasks if the colorimetric determination is not performed immediately.

5.0 EQUIPMENT AND SUPPLIES

- 50ml volumetric flasks
- 50 or 100ml class A graduated cylinders
- UV/Vis spectrophotometer, 1 cm or longer pathlength, capable of measurement at 578nm
- Fume hood
- Eppendorf and Oxford pipettors (calibrated)

6.0 REAGENTS

- ASTM Type II (or better) water should be used in the preparation of all standards and reagents.

- All chemicals must be ACS reagent grade

- All reagents and standards must be prepared in class A volumetric glassware

6.1 Manual Colorimetric Reagents:

- Phosphate Buffer: Dissolve 138 grams of $NaH_2PO_4 \cdot H_2O$ in water and dilute to1 liter. Keep refrigerated. Prepare fresh monthly. Unused/expired solution can be disposed of down the drain.

- Chloramine-T solution: Dissolve 1.0 gram chloramine-T in water and dilute to 100ml. Prepare fresh daily. Unused solution can be disposed of down the drain.

- Pyridine-barbituric acid color reagent: Prepare in a fume hood. Place 15 grams barbituric acid in a 500ml volumetric flask. Rinse down the sides of the flask with just enough water to wet the acid. Add a magnetic stir bar and place flask on a magnetic stir plate. Add, with stirring, 75ml pyridine and 15ml concentrated HCl. Slowly add 300ml water. Stir until barbituric acid has dissolved. Dilute to 500ml. Store in a dark bottle in the refrigerator. This solution is good for 6 months. Unused solution should be disposed of in the Non-Chlorinated Waste container.

- 0.25N NaOH: Dissolve 20g sodium hydroxide pellets in di water and dilute to 2 liters. This solution expires one year after preparation. Unused/expired solution can be neutralized (pH 5-8) and disposed of down the drain.

6.2 Stock Standards:

- 1000mg/L CN stock standard, commercially available with certification and expiration date. Unused/expired standard should be disposed of in the Non-Chlorinated Waste container.

- A separate source free cyanide standard is used for calibration verification (ICV/CCV). Unused/expired standard should be disposed of in the Non-Chlorinated Waste container.

7.0 PROCEDURE

7.1 Turn on the UV/Vis spectrophotometer and set the wavelength to 578nm. Allow to warm up for one hour.

7.2 Prepare a series of standards for a calibration curve from a 10mg/L CN working solution prepared from the CN stock standard:

standard conc (ug/L)	ml working sol/25ml NaOH
0	0
5	0.0125
20	0.050
50	0.125
50	0.250
150	0.375
200	0.500

7.3 Add 25ml of standard or sample to a 50ml volumetric flask, or a smaller portion of sample diluted to 25ml with 0.25N NaOH.

7.4 Add 8.0ml of phosphate buffer and mix

7.5 Add 1.0ml chloramine-T solution and mix. Wait 1 - 2 minutes.

7.6 Add 2.5ml pyridine-barbituric acid color reagent and mix.

7.7 Bring the sample/standard up to 50ml volume **with DI water**, and mix.

7.8 Allow to develop for 8 minutes, then read absorbance at 578nm within 15 minutes.

7.9 Zero the spectrophotometer with a reagent blank. Pour a portion of the calibration blank into the cell and adjust the absorbance to .000. Remove the cell, wait 10-20 second and replace it. If the reading is not still .000, readjust and repeat. Read and record all standard/sample absorbances and required dilutions in the Colorimetric Logbook.

7.10 All waste from the colored samples and rinsing should be disposed of in the Non-Chlorinated Waste container.

7.11 Plot the calibration curve using linear regression with the standard absorbance vs. concentration. The correlation coefficient must be 0.995 of greater and the y-intercept must be less than the reporting limit.

7.12 Calculate sample concentrations in ug/L by comparing sample absorbances to the calibration curve. If any sample has an absorbance above the calibrated range, repeat the colorization process with an appropriate volume of distillate diluted to 25ml with 0.25N NaOH.

7.13 For soil and sediment samples, determine the cyanide concentration of the solid sample as follows:

$$\text{Dry basis:} \quad \text{cyanide, mg/kg} = \frac{(x)\,(y)\,(1{,}000{,}000\ \text{ug/Kg})}{g\ (\%S)}$$

Where:

x = cyanide concentration in distillate, ug/L
y = final extract volume, liters
g = wet weight of solid sample, g
%S = percent solids in solid sample (as decimal fraction)

7.14 Record all standard/sample absorbances and required dilutions in the Cyanide Analysis Logbook.

8.0 QUALITY CONTROL

8.1 The ICV/CCV standard should be prepared from a free cyanide source different than that from which the calibration curve was prepared. The ICV standard must be analyzed immediately following the calibration before the analysis of the samples. The ICV standard should be prepared or diluted such that the concentration is near the mid-point of the curve. The percent recovery must be 90 - 110%. A CCV must be analyzed after every 10 samples. Recovery must be 90 - 110%. If a CCV fails these limits, all samples after the last passing CCV must be reanalyzed.

8.2 An ICB/CCB must be analyzed following every ICV/CCV. The ICB/CCB samples must be less than half of the reporting limit.

8.3 One sample duplicate must be distilled and analyzed with every batch of 20 samples, or less, per matrix. The sample and duplicate concentration RPD must be less than 15%. If the RPD exceeds this limit, the sample and duplicate should be re-distilled and the cause of the possible contamination or interference should be investigated.

8.4 One matrix spike sample must be distilled and analyzed with every batch of 20 samples, or less, per matrix. Matrix spike must be added to the sample before any step in the distillation process is begun. The spike concentration should be approximately 40ug/L. Spike recovery should be 85-115%. If the recovery is outside these limits, check for matrix interference by analyzing a post-distillation spike.

8.5 A prep blank, low calibration standard (20ug/L), mid-range calibration standard (100ug/L) and second source LCS standard are distilled to be analyzed along with each sample batch. The prep blank concentration must be less than half of the reporting limit. The low and mid-range distilled standard concentrations must have a recovery of 90-110%. The LCS standard must have a recovery of 85-115%. If any of these QC fail to meet the requirement, the batch must be re-distilled.

9.0 METHOD DETECTION LIMITS

9.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit official book.

10.0 METHOD PERFORMANCE

10.1 The MDL concentrations listed in the GPL MDL book are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year per analyst.

11.0 POLLUTION PREVENTION

11.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

12.0 SAFETY

12.1 The analysis should be performed under a fume hood. Pyridine is aromatic and a carcinogen.

12.2 Keep all samples and standards away from acids to avoid liberating toxic HCN gas.

12.3 Wear safety glasses, lab coat, and gloves when handling samples and reagents.

12.4 Use special care when handling strong acids and NaOH solutions.

13.0 DISPOSAL REQUIREMENTS

13.1 All waste should be collected from the analysis in a bottle for Cyanide Waste only. Label the bottle: Cyanide waste; contains pyridine. More details concerning disposal characteristics and procedures can be located in the SOP D.1 " Laboratory Waste Handling and Disposal Procedure".

14.0 REPORTING REQUIREMENTS

14.1 A LIMS batch must be generated for each analytical run. A copy of the Cyanide Analysis Logbook pages and the Cyanide Preparation Log must be attached to the report

14.2 QC records are maintained in the form of control charts to document percent recovery of cyanide from laboratory control samples subjected to the distillation procedure. See SOP E.4, "Quality Control Charts" for more information.

15.0 DEFINITIONS

15.1 For definition of terminologies used in this document, refer to GPL Laboratories SOP G.14.

SOP No: J.13

Title: Analysis of Waste Liquid and Solid Samples for Reactivity as Defined by SW846 Chapter 7.

Scope: This Standard Operating Procedure describes the method for determination of the reactivity of waste samples as required by SW846.

1.0 PURPOSE

1.1 This procedure describes the methodology used to determine the reactivity of samples as defined in 40 CFR 261.23 and is designed to identify wastes that consist of the following properties:

- Readily undergo violent chemical change;
- React violently or form potentially explosive mixtures with water;
- Generate toxic fumes when mixed with water, or, in the case of cyanide- or sulfide-bearing wastes, when exposed to mild acidic of basic conditions.

This SOP and method are intended only to measure cyanide and sulfide as evolved under the test conditions.

2.0 REFERENCES

- SW846 Chapter 7
- GPL SOP's for CN (J.43) and S_2 (J.11) determination

3.0 EQUIPMENT AND SUPPLIES

- 500ml round bottom, 2-neck flask
- 50ml gas scrubber, glass joint to fit 2-neck flask
- Stir plate and teflon coated stir bar
- Addition funnel, glass joint to fit 2-neck flask
- Flexible tubing and gas flow meter
- Source of Nitrogen gas

4.0 REAGENTS

4.1 0.01N Sulfuric Acid Solution – Add 0.272ml concentrated Sulfuric Acid to di water in a 1 liter flask and dilute to volume. This solution expires after one year. Unused/expired solution should be disposed of in the Acid Waste container.

4.2 0.25N Sodium Hydroxide Solution – dissolve 10g sodium hydroxide pellets in di water with stirring and dilute to 1 liter. This solution expires one year after preparation. Unused/expired solution can be neutralized (pH 5-8) and disposed of down the drain.

4.3 Cyanide reference stock standard – 1000mg/L certified solution is purchased. Unused/expired standard must be disposed of in the Non-Chlorinated Waste container.

4.4 Sulfide reference stock standard – a certified solution can be purchased from ERA. Alternatively, a solution can be made using sodium sulfide nonahydrate that has been washed and blotted dry. Approximately 6g of sodium sulfide should be weighed immediately and dissolved. The value of the fresh solution should be around 800mg/L. The true value of the solution for recovery calculations should be determined daily by analyzing an undistilled portion. This solution expires one year after preparation, or if the determined true value falls below 400mg/L. Unused/expired standard should be disposed of in the Non-Chlorinated Waste container.

5.0 PROCEDURE

5.1 Weigh out approximately 10 grams of the sample to be analyzed. Record the exact weight in the Reactivity Prep Logbook. Record the weight of water and liquid waste samples as well as the volume (10ml) used. Transfer the sample into the 2-neck flask.

5.2 Add 50ml of NaOH solution to the gas scrubber.

5.3 Connect the distillation apparatus as in figure 1.

5.4 Add 250ml of the 0.01N sulfuric acid solution to the addition funnel (with the stopcock closed) and cap the funnel with the gas inlet. Adjust the Nitrogen flow until the gas scrubbers begin bubbling.

5.5 Allow the system to purge with nitrogen for 10 minutes.

5.6 Open the stopcock on the addition funnel. Begin stirring while the acid is entering the flask. Stirring speed must remain constant during the test period. The stirring should be performed at a slow rate that will not create a vortex, but quickly enough to keep any solid material suspended. Maintain the stirring and the gas bubbling in the scrubbers for 30 minutes.

5.7 After 30 minutes, close off the nitrogen and disconnect the scrubber.

5.8 Dispose of the waste sample left in the flask in the Acid Waste container.

5.9 Determine the amount of cyanide and sulfide released into the scrubber liquid by:

5.9.1 CN - SOP J.43

5.9.2 S_2 - SOP J.11

6.0 QUALITY CONTROL

6.1 A blank sample will be run through the entire procedure, with CN and Sulfide results BQL.

6.2 A sample with a known amount of cyanide and sulfide will be distilled. For cyanide, 1ml of the 1000mg/L stock standard is used. For sulfide, 10ml of the stock standard are used. The cyanide and sulfide standards must be distilled separately to avoid interference in the cyanide analysis. Results for these LCS samples are typically low because of the test conditions. Recovery limits are statistically determined from previous analyses.

6.3 One sample duplicate must be extracted for every batch of 20 samples or less, per matrix.

7.0 SAFETY

7.1 Care should be taken when handling reagents and samples. A lab coat, safety glasses and gloves should be worn while performing the analyses.

7.2 Keep all samples and standards away from acids to avoid liberating toxic HCN gas.

8.0 DISPOSAL REQUIREMENTS

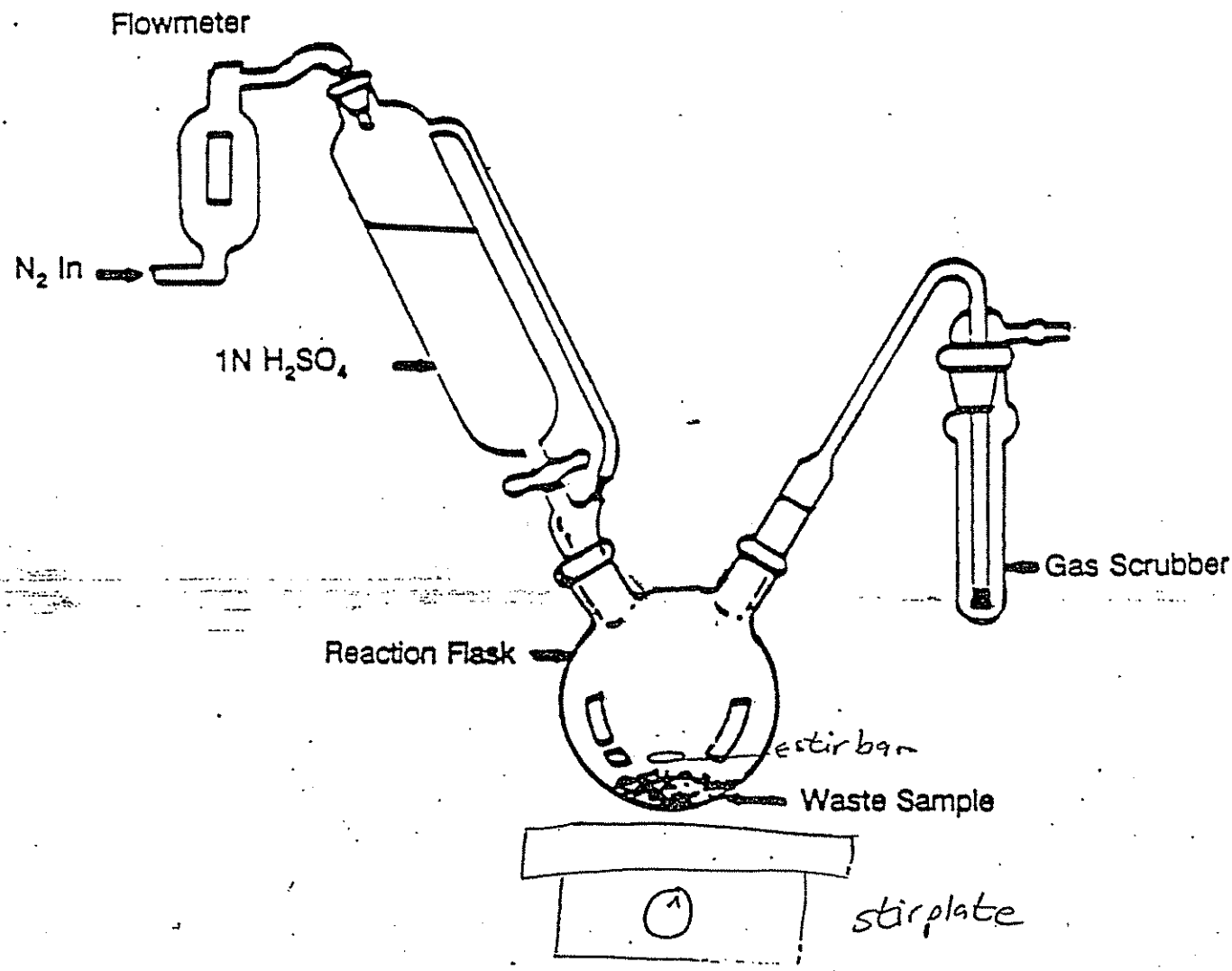
8.1 Details concerning disposal characteristics and procedures can be located in the SOP D.1 "Laboratory Waste Handling and Disposal Procedure".

9.0 DEFINITIONS

9.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

FIGURE 1

APPARATUS TO DETERMINE HYDROGEN CYANIDE AND SULFIDE RELEASED FROM WASTES



SW846

SOP No: J.12

Title: Analysis of Waste Liquid and Solid Samples for pH, and Corrosivity as pH as Defined by SW846 Volume IC, Chapter 7. (7.2.2 - 1.a only), using Method 9045

Scope: This Standard Operating Procedure describes the method for determining sample pH. It also describes the determination of corrosivity in waste samples as required by SW846.

1.0 PURPOSE

1.1 This procedure describes the methodology used to determine the pH of solid samples using EPA method 9045. The procedure can also be used to determine the corrosivity of samples as defined in 40 CFR 261.22. This document is designed to identify wastes that may pose a hazard to either human health or the environment due to their ability to:

- Mobilize toxic metals if discharged into a landfill;
- Corrode handling, storage, and transportation equipment;
- Destroy human or animal tissue with contact.

In order to identify such potentially hazardous materials, EPA has selected two properties upon which to base the definition of a corrosive waste. These properties are pH and corrosivity toward Type SAE 1020 steel.

This SOP deals only with the first corrosive property, pH.

2.0 REFERENCES

- SW846 Vol. IC, Chap. 7
- SW846 Method 9045
- GPL SOP for pH determination

3.0 EQUIPMENT AND SUPPLIES

3.1 50ml plastic, disposable centrifuge tubes

3.2 analytical balance

3.3 pH meter

3.4 Combination pH/ATC electrode

3.5 Thermometer if automatic temperature compensation (ATC) probe is not used

3.6 Buffers at pH 2.00, 4.00, 7.00, 10.00, and 12.0

4.0 PROCEDURE

4.1 For liquid samples refer directly to pH SOP No. J.5.

4.2 For solid samples refer directly to pH SOP No. J.5 Section 4.3.6

5.0 QUALITY CONTROL REQUIREMENTS

Quality Control requirements for pH analysis consist of periodic verification of standard pH buffers to determine instrument performance and to detect electrode drift.

5.1 After the meter is initially calibrated, a calibration verification (pH 7 buffer) is analyzed. After every ten sample analyses a set of standard pH buffers are analyzed which bracket the pH range of the samples that have been analyzed. For most sample types, pH 4 and 7, and pH 7 & 10 alternated should cover the sample range. For example; ICV = pH 7, CCV1 = pH 4 and 7, CCV2 = pH 7 and 10, CCV3 = pH 4 & 7, etc. For samples with pH above 10, a pH 12 buffer should be checked to verify that the probe is linear above 10. Similarly, if samples are analyzed that have pH less than 4, a pH 2 buffer should be analyzed for a CCV. If the obtained value deviates more than ± 0.1 pH units from the known value the instrument shall be recalibrated and the last ten samples reanalyzed.

5.2 A duplicate analysis must be performed for every 20 or fewer samples of the same matrix. At least one duplicate must be performed per day. The sample and duplicate pH readings should not differ by more than 0.10 pH units. If the difference between the readings differs by more than this, the sample and duplicate analyses must be repeated.

5.3 Analysis of blanks and spikes are not applicable to this parameter.

6.0 DATA RECORDING

6.1 Instrument Log

An Instrument Log shall be maintained for each pH meter in use. The Log shall be used to record the date the instrument was used, the name of the person using the instrument, the buffers used to calibrate the instrument, any additional calibrations required and any problems encountered. Additionally, maintenance of the pH probe(s) shall be recorded in the Log.

6.2 Data Recording

Data obtained from pH analyses shall be recorded in the pH Data Book . Data recording practices shall conform with SOP.

7.0 SAFETY

7.1 Wear safety glasses, lab coat, and gloves when handling samples.

8.0 WASTE DISPOSAL

8.1 See GPL Laboratories SOP D.1 "Analytical Division Laboratory Waste Handling and Disposal Procedure."

9.0 DEFINITIONS

9.1 For definition of terminologies used in this document, refer to GPL Laboratories SOP G.14

SOP No: J.13

Title: Analysis of Waste Liquid and Solid Samples for Reactivity as Defined by SW846 Chapter 7.

Scope: This Standard Operating Procedure describes the method for determination of the reactivity of waste samples as required by SW846.

1.0 PURPOSE

1.1 This procedure describes the methodology used to determine the reactivity of samples as defined in 40 CFR 261.23 and is designed to identify wastes that consist of the following properties:

- Readily undergo violent chemical change;
- React violently or form potentially explosive mixtures with water;
- Generate toxic fumes when mixed with water, or, in the case of cyanide- or sulfide-bearing wastes, when exposed to mild acidic of basic conditions.

This SOP and method are intended only to measure cyanide and sulfide as evolved under the test conditions.

2.0 REFERENCES

- SW846 Chapter 7
- GPL SOP's for CN (J.43) and S_2 (J.11) determination

3.0 EQUIPMENT AND SUPPLIES

- 500ml round bottom, 2-neck flask
- 50ml gas scrubber, glass joint to fit 2-neck flask
- Stir plate and teflon coated stir bar
- Addition funnel, glass joint to fit 2-neck flask
- Flexible tubing and gas flow meter
- Source of Nitrogen gas

4.0 REAGENTS

4.1 0.01N Sulfuric Acid Solution – Add 0.272ml concentrated Sulfuric Acid to di water in a 1 liter flask and dilute to volume. This solution expires after one year. Unused/expired solution should be disposed of in the Acid Waste container.

4.2 0.25N Sodium Hydroxide Solution – dissolve 10g sodium hydroxide pellets in di water with stirring and dilute to 1 liter. This solution expires one year after preparation. Unused/expired solution can be neutralized (pH 5-8) and disposed of down the drain.

4.3 Cyanide reference stock standard – 1000mg/L certified solution is purchased. Unused/expired standard must be disposed of in the Non-Chlorinated Waste container.

4.4 Sulfide reference stock standard – a certified solution can be purchased from ERA. Alternatively, a solution can be made using sodium sulfide nonahydrate that has been washed and blotted dry. Approximately 6g of sodium sulfide should be weighed immediately and dissolved. The value of the fresh solution should be around 800mg/L. The true value of the solution for recovery calculations should be determined daily by analyzing an undistilled portion. This solution expires one year after preparation, or if the determined true value falls below 400mg/L. Unused/expired standard should be disposed of in the Non-Chlorinated Waste container.

5.0 PROCEDURE

5.1 Weigh out approximately 10 grams of the sample to be analyzed. Record the exact weight in the Reactivity Prep Logbook. Record the weight of water and liquid waste samples as well as the volume (10ml) used. Transfer the sample into the 2-neck flask.

5.2 Add 50ml of NaOH solution to the gas scrubber.

5.3 Connect the distillation apparatus as in figure 1.

5.4 Add 250ml of the 0.01N sulfuric acid solution to the addition funnel (with the stopcock closed) and cap the funnel with the gas inlet. Adjust the Nitrogen flow until the gas scrubbers begin bubbling.

5.5 Allow the system to purge with nitrogen for 10 minutes.

5.6 Open the stopcock on the addition funnel. Begin stirring while the acid is entering the flask. Stirring speed must remain constant during the test period. The stirring should be performed at a slow rate that will not create a vortex, but quickly enough to keep any solid material suspended. Maintain the stirring and the gas bubbling in the scrubbers for 30 minutes.

5.7 After 30 minutes, close off the nitrogen and disconnect the scrubber.

5.8 Dispose of the waste sample left in the flask in the Acid Waste container.

5.9 Determine the amount of cyanide and sulfide released into the scrubber liquid by:

5.9.1 CN - SOP J.43

5.9.2 S_2 - SOP J.11

6.0 QUALITY CONTROL

6.1 A blank sample will be run through the entire procedure, with CN and Sulfide results BQL.

6.2 A sample with a known amount of cyanide and sulfide will be distilled. For cyanide, 1ml of the 1000mg/L stock standard is used. For sulfide, 10ml of the stock standard are used. The cyanide and sulfide standards must be distilled separately to avoid interference in the cyanide analysis. Results for these LCS samples are typically low because of the test conditions. Recovery limits are statistically determined from previous analyses.

6.3 One sample duplicate must be extracted for every batch of 20 samples or less, per matrix.

7.0 SAFETY

7.1 Care should be taken when handling reagents and samples. A lab coat, safety glasses and gloves should be worn while performing the analyses.

7.2 Keep all samples and standards away from acids to avoid liberating toxic HCN gas.

8.0 DISPOSAL REQUIREMENTS

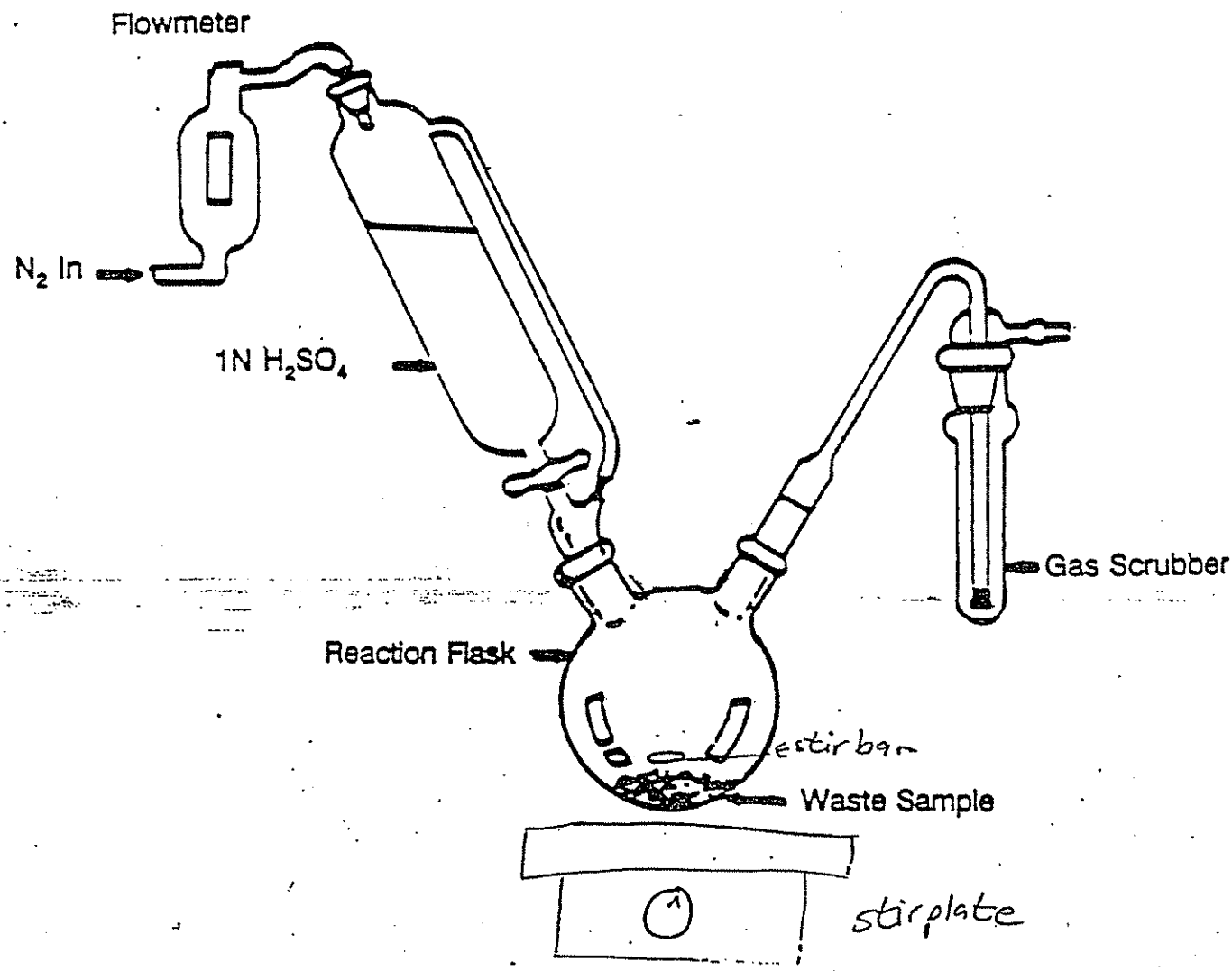
8.1 Details concerning disposal characteristics and procedures can be located in the SOP D.1 "Laboratory Waste Handling and Disposal Procedure".

9.0 DEFINITIONS

9.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

FIGURE 1

APPARATUS TO DETERMINE HYDROGEN CYANIDE AND SULFIDE RELEASED FROM WASTES



SW846

SOP No: J.11

Title: Analysis of Water Samples and Soil Distillates for Sulfide According to MCAWW Method 376.1, SW846 9030 and 9034 (titrimetric)

Scope: This Standard Operating Procedure describes the method for determination of sulfide for water samples and soil distillates as required by MCAWW Method 376.1 and SW846 methods 9030 and 9034 (titrimetric)

1.0 PURPOSE

1.1 This procedure describes the methodology used to determine the sulfide content of samples according to the method described in the Methods for Chemical Analysis of Water and Wastes (MCAWW), Method 376.1 (Titrimetric) and SW864 methods 9030 and 9034. The methodology is applicable to drinking, surface, and saline waters, domestic and industrial washes. Distillates of soil, solid and waste samples and Reactive Sulfide distillates are also analyzed by this SOP.

2.0 REFERENCES

- Methods for Chemical Analysis of Water and Wastes (MCAWW), EPA-600/4-82-055, Method 376.1 (Titrimetric)
- SW846 Methods 9030 and 9034 (Titrimetric)

3.0. EQUIPMENT AND SUPPLIES

- 10ml buret, accurate to 0.05ml
- 125ml glass Erlenmeyer flask or beaker
- Electronic stir plate and magnetic stir bar

4.0 REAGENTS

- Sodium Hydroxide, 6N – Dissolve 240g sodium hydroxide pellets in di water with stirring and dilute to 1 liter. This solution is used for sample preservation. This solution is good for one year. Unused/expired solution can be neutralized (pH 5-8) and disposed of down the drain.

- Zinc Acetate solution, 2N – Dissolve 220g zinc acetate dihydrate in di water and dilute to 500ml. This solution is used for sample preservation. This solution expires after one year. Unused/expired solution should be disposed of in the Non-Chlorinated Waste container.

- Hydrochloric Acid, 6N - Carefully add 500ml of concentrated hydrochloric acid to 500ml of di water. Solution will be hot. This solution expires after one year. Unused/expired solution should be disposed of in the Acid Waste container.

- Standard iodine solution, 0.0250N – Dissolve 20g Potassium Iodide crystals in di water and dilute to 1 liter. Standardize daily versus Standard Sodium Thiosulfate solution. This solution expires after one year. Dispose of any unused/expired solution down the drain.

- Standard sodium thiosulfate solution, 0.0250N – A certified standard solution is purchased. If the solution is not provided with an expiration date by the manufacturer, a date of one year from receipt must be used. Dispose of any unused/expired solution down the drain.

- Starch indicator solution – 1% w/v solution is purchased. Unused/expired solution should be disposed of in the Acid Waste container.

5.0 PROCEDURE

5.1 Sample holding time and preservation

5.1.1 Water samples must be analyzed within 7 days of collection if zinc acetate is added as a preservative. Unpreserved samples must be analyzed immediately. Soil sample distillates should be analyzed immediately.

5.2 Standardization of iodine solution

5.2.1. Add 5.0ml of the standard iodine solution to 20ml of laboratory pure water. Acidify by adding 2.0ml of 6N HCl. Add a few drops of starch indicator to form a blue or black color.

5.2.2. Titrate using the certified standard sodium thiosulfate solution. As the end-point is near, the color will change from black to blue. Titrate slowly from this point. Titrate until the blue color disappears completely.

5.2.3. Record the amount of titrant used.

5.2.4. Repeat this procedure a second time and calculate the average normality

$$\text{Iodine N} = \frac{(\text{ml titrant used})(0.025\text{N})}{5 \text{ ml iodine}}$$

5.3 Sample analysis

5.3.1 Add 5.0ml of iodine solution and 2.0ml 6N HCl to 20ml di water in the titrating flask. (if distillates for acid-insoluble sulfide as being analyzed, use 10ml of 6N HCl)

5.3.2 Introduce 20ml of sample beneath the level of the iodine using a disposable serological pipet. If the Iodine color is reduced to pale yellow, begin again using 10ml, or a volume of Iodine solution to produce the amber color expected.

5.3.3 Add a few drops of starch indicator to form a black or dark blue color.

5.3.4 Titrate as above, with standardized sodium thiosulfate solution. Record the volume of titrant used.

5.3.5 Titrated samples can be disposed of in the Acid Waste container.

5.4 Soil samples

5.4.1 Soil and waste samples should be distilled for acid-soluble and/or acid insoluble sulfides, as appropriate, by method 9030B prior to analysis. See SOP J.63.

5.5 Calculation

5.5.1 $$\text{Sulfide, mg/L} = \frac{[(A \times B) - (C \times D)] \times 16{,}000}{\text{ml sample}}$$

where:

A = ml of iodine solution added
B = Normality of iodine solution
C = ml of titrant used
D = Normality of titrant used

6.0 QUALITY CONTROL

6.1 A purchased, certified Sulfide standard is used as the ICV/CCV solution. The ICV is analyzed before all sample analysis. A CCV is analyzed after every 10 or fewer samples and at the end of the analytical run. Recovery limits provided with the certified standard will be applied.

6.2 Control blanks will be analyzed at the beginning of the analysis, and continuing after every ten samples, as well as at the end of the analysis.

6.3 A sample duplicate analysis will be performed on one out of every 20 or fewer samples of each matrix. The sample/duplicate RPD must be less than 15%.

6.4 A matrix spiked analysis will be performed on one out of every 20 or fewer samples to determine matrix effects.

7.0 SAFETY

7.1 Care should be taken when handling reagents and samples.

SOP No: H.10

Title: Trace ICP Quantitation of HSL Metals plus Boron, Molybdenum, Silicon, Strontium, Titanium, and Tin According to Method 6010B

Scope: The method detailed in this procedure is for the analysis of water, TCLP and EP extracts, soils, sludges, sediments and other solid wastes digestates for Hazardous Substance List (HSL) Metals by Inductively Coupled Plasma (ICP) spectroscopy in accordance with USEPA method 6010B. The use of ionization buffers, internal standards, and special background correction techniques is specified.

1.0 INTERFERENCES

1.1 There are four main categories of interferences: unresolved overlap of molecular band spectra, stray light, overlap from nearby spectral lines, and background emission from continuous or recombination phenomena.

2.0 PURPOSE

2.1 The purpose of this procedure is to describe the simultaneous analysis of metals on the USEPA Hazardous Substances List (Antimony, Arsenic, Lead, Selenium, Silver, Thallium, Sodium, Potassium, Aluminum, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Magnesium, Manganese, Nickel, Vanadium, and Zinc) plus Boron, Molybdenum, Strontium, Titanium, and Tin at trace levels using a Thermo-Jarrell-Ash 61E Purged Trace Inductively Coupled Plasma Spectrometer and autosampler. All samples are digested in accordance with SOP H.4 or H.5 prior to analysis. Filtered samples for dissolved metals analysis can be analyzed after either digestion or matrix matching. The digestate holding time is 180 days.

2.2 Prior to use this method, the samples should be prepared using appropriate sample preparation methods (SOP#H8 – 3010, SOP#H21 – 3050)

3.0 REFERENCES

- TJA ICAP 61E Operator's Manual (p/n 134542-00).
- SW846 method 6010B revision 1.

4.0 CALIBRATION PARAMETERS

Element	Wavelength	Element	Wavelength
Ag	3280	Mo	2020
Al	3082	Na	3302
As	1890	Ni	2316
B	2496	Pb	2203
Ba	2347	Se	1960
Be	3130	Sb	2068
Ca	3179	Si	2881
Cd	2265	Sn	1899
Co	2286	Sr	4125
Cr	2677	Ti	3349
Cu	3247	Tl	1908
Fe	2714	V	2924
K	7664	Y	3710
Mg	2790	Zn	2062
Mn	2576		

5.0 EQUIPMENT AND SUPPLIES

- 100mL volumetric flasks (Class A)
- 200mL volumetric flasks (Class A)
- 500mL volumetric flasks (Class A)
- 1000mL volumetric flasks (Class A)
- 100mL plastic storage bottles
- 250mL plastic storage bottles
- 500mL plastic storage bottles
- 1000mL plastic storage bottles
- 20.0L Nalgene carboy
- 10.0L Nalgene carboy
- 15mL disposable autosampler tubes
- 28mL disposable autosampler tubes
- Centrifuge tube holder
- Pipetters - Wheaton calibra M25 Finnpipette II M26
- Pipette Tips
- 5, 10, 20mL Class A volumetric pipets
- Pump windings and tee fittings
- Argon gas (cryogenic liquid source)
- Nitrogen gas (cryogenic liquid source) used for purging of spectrometer

5.1 Instrumentation

5.1.1 TJA 61E Purge Trace ICP with meinhard nebulizer, cyclonic spray chamber, horizontal torch, and AS-192 autosampler.

5.1.2 Simultaneous background correction technique is used for the analysis of lead and selenium to achieve lower instrumental detection limits comparable to graphite furnace.

5.1.3 Yttrium Internal standard - Lithium ionization buffer is added on-line using a mixing tee and coil with a ratio of 1:4 (one part standard: four parts samples, resulting in a dilution factor of 5 for the internal standard, approximated 10ppm is the final "mixed-in" concentration.

6.0 REAGENTS

- A deionized water ASTM type II or equivalent
- Concentrated hydrochloric acid, trace metals grade
- Concentrated nitric acid, trace metals grade
- Flame Water - 5% hydrochloric acid, 1% nitric acid

Preparation of Reagent Flame Water

Fill a 20L Nalgene carboy half full with type II water. Add 200ml concentrated nitric acid and 1000ml concentrated hydrochloric acid underneath a hood to contain noxious gases. Dilute to twenty liters with type II water and mix thoroughly.

2% nitric acid

5ppm Arsenic Profile check solution

Preparation of Arsenic Solution:

Pipette 2.5mL of 1000ppm Arsenic stock solution into a 500mL volumetric flask. Dilute to volume with matrix matched water. Transfer to a bottle labeled 5ppm As. Record the date of preparation, expiration date and preparers initials on the bottle label. Prepare Arsenic Profile solution every three months or when depleted, whichever is more frequent.

50ppm Y-1500ppm lithium internal standards ionization buffer

Preparation of 50ppm Y-1500ppm Lithium Internal standard-ionization buffer:

Pipet 100mls of GP mix Li into 1000ml volumetric flask. Dilute to volume with flame water.

All stock and prepared standards are logged into a software program (solutions manager). All stock and prepared standards are assigned a solutions manager reference number. The reference number expiration date, prep date and chemist initials are written on a label and placed on all prepared solutions. The expiration date chosen by solutions manager for prepared standards is three months for the prepared date.

7.0 GENERAL PRECAUTIONS

7.1 AVOID CONTAMINATION of STOCK STANDARDS. Always pour a small volume of standard stock solution into a new microbeaker before pipetting an aliquot. NEVER insert a pipette directly into the bottle. This precautionary measure also applies to quality control standards stock solutions (ICVA, IC1, etc.)

7.2 Check pipetters daily for leaks and proper calibration. Record in the pipette log book.

7.3 Empty Drain vessel at the end of each day. Transport waste to the waste disposal area for appropriate treatment prior to shipment. Fill vessel up to 6 inches using tap water prior to replacing vessel beneath instrument.

7.4 Clean up and neutralize all spills immediately to avoid corrosion damage to the instrument.

7.5 Purge the optics with nitrogen at all times. Leave the RF power unit on at all times. Never attempt to perform repairs to the High voltage systems. Leave the instrument PM tubes and heater on at all times.

8.0 PROCEDURE

8.1 Preparation of Calibration Standards

8.1.1 To prepare calibration Standard 1 in flame water, pipette 25mL of source solution Sp Mx Std. 12 into a 500mL flask, which has been half filled with flame water. Dilute the flask to volume with flame water. Mix the solution thoroughly, and transfer it to a plastic bottle.

True value of Standard 1

Element	Certified Std. Conc.(ug/ml) Added	Volume (ml)	Calibration Std. Conc. (ug/L)
Aluminum	1000	25	50000
Antimony	10	25	500
Arsenic	10	25	500
Barium	10	25	500
Beryllium	1.000	25	50
Boron	10	25	500
Cadmium	10	25	500
Chromium	10	25	500
Cobalt	10	25	500
Copper	10	25	500
Iron	1000	25	50000
Lead	10	25	500
Manganese	10	25	500
Molybdenum	10	25	500
Nickel	10	25	500
Selenium	10	25	500
Silicon	10	25	500
Silver	10	25	500
Strontium	10	25	500
Thallium	10	25	500
Tin	10	25	500
Titanium	10	25	500
Vanadium	10	25	500
Zinc	10	25	500

8.1.2 To prepare calibration Standard 2 in flame water, pipette 50mL of source solution Sp Mx Std. 12 into a 500mL flask, which has been half filled with flame water. Dilute the flask to volume with flame water. Mix the solution thoroughly, and transfer it to a plastic bottle.

True value of Standard 2

Element	Certified Std. Conc.(ug/ml) Added	Volume (ml)	Calibration Std. Conc. (ug/L)
Aluminum	1000	50	100000
Antimony	10	50	1000
Arsenic	10	50	1000
Barium	10	50	1000
Beryllium	1.000	50	100
Boron	10	50	1000
Cadmium	10	50	1000
Chromium	10	50	1000
Cobalt	10	50	1000
Copper	10	50	1000
Iron	1000	50	100000
Lead	10	50	1000
Manganese	10	50	1000
Molybdenum	10	50	1000
Nickel	10	50	1000
Selenium	10	50	1000
Silicon	10	50	1000
Silver	10	50	1000
Strontium	10	50	1000
Thallium	10	50	1000
Tin	10	50	1000
Titanium	10	50	1000
Vanadium	10	50	1000
Zinc	10	50	1000

8.1.3 To prepare calibration Standard 3 in flame water, add 10ml each of the Mg, Ca, Na stock (10,000ppm) and 1.250ml of K stock standard (10,000ppm) standard from High Purity standard with a Class A volumetric pipets to a 500mL volumetric flask which has been half filled with flame water. Dilute the flask to volume with flame water, mix the solution thoroughly, and transfer to a plastic bottle.

Standard 3	Starting CONC	Volume Added	Final CONC.
Sodium	10,000ug/ml	10ml	200000ug/L
Potassium	10,000ug/ml	1.25ml	25000ug/L
Magnesium	10,000ug/ml	10ml	200000ug/L
Calcium	10,000ug/ml	10ml	200000ug/L

NOTE 1: These formulations are subject to change without an update of SOP to suit various client requirements

NOTE 2: A multipoint calibration is performed daily for those elements in standards 1 and 2. Standard 3 is used to perform a five point calibration that is re-sloped daily.

8.1.4 Document the standard in solutions manager program. Label the standard solution bottle. The label should include the preparers initials, the date of preparation, the expiration date, and the page number on which the standard has been recorded in the log book. The expiration date of standard solutions is three months from the date of preparation or whenever one of the certified standards expires, whichever is first.

8.2 QC Preparation

8.2.1 Preparation of Low level linearity check Solution.(PQL)

Pipette appropriate volume of single element STP's into 1000ml volumetric flask. Fill to volume with flame water.

Note: Additional elements can be added, if necessary, according to client needs.

Document the solution in the Solutions Manager Software Program. Label the bottle as ICP Stock and record the preparation date, the expiration date, the preparers initials, and the ID number.

8.2.2 The CCV is made from the stock solutions used in making the calibration standards. The CCV concentrations are at half the concentrations of the highest standards used in calibration.

8.2.3 Initial Calibration Verification (ICV)

Fill a 1L volumetric flask halfway with flame water and add ten mLs of ICV, certified stock solution. Dilute the flask to volume with flame water. Mix the solution thoroughly and transfer to a plastic bottle labeled ICV.

8.2.4 Interference Check Standard A (ICSA):

Fill a 500mL volumetric flask halfway with flame water and add 50mL of ICSA certified multi-element stock solution. Dilute the flask to volume with flame water. Mix the solution thoroughly and transfer to a plastic bottle labeled ICSA.

8.2.5 Interference Check Standard AB (ICSAB):

Fill a 500mL volumetric flask halfway with flame water and add 50mL of ICSA certified stock solution, and 5mL of SM-421-012 and 10mL of CLP calibration mix #2. Dilute the flask to volume with flame water. Mix the solution thoroughly and transfer to a plastic bottle labeled ICSAB.

8.3 Preparation of 1:5 Serial Dilution (L)

Obtain the sample digestates for the case or SDG to be analyzed. Take the original sample digestate that corresponds to the sample designated for duplicate and matrix spike digestions for each SDG and matrix and prepare its serial dilution as follows:

Transfer 2mls of sample into 8mls flame water. Mix thoroughly.

8.4 Preparation of Post Digestion Spike

Obtain the sample digestates for the batch or SDG analyzed. Take the original sample digestate that corresponds to the sample designated for duplicate and matrix spike digestions for each batch and prepare the post spike as follows.

Transfer 10ml of sample into a sample tube. Remove 0.1mls of sample, then spike with 0.1ml of post spike solution. Mix thoroughly.

Note: If the matrix spike recovery is outside criteria, the sample may require spiking at alternate concentrations

8.5 Tuning and Calibration of the ICP

8.5.1 Conduct a pre-start up inspection.

8.5.1.1 Argon and Nitrogen gas supply and drain vessel

Make sure there is an adequate supply of Argon. The argon line pressure regulator should be set at 60psi and the nitrogen line pressure should be set at 50psi.

Check the drain vessel beneath the ICP and empty it if full.

8.5.1.2 Torch box

Make sure all connections are secure and air tight, including the drain hose, nebulizer cap, argon lines.

8.5.1.3 Peristaltic pump

Install new flexible pump tubing every other day (the windings have three stops which allow for an extra day of use) or if the old one shows signs of flattening or stretching, and connect to the nebulizer with capillary tubing.

8.5.2 Daily Start Up

8.5.2.1 Reset computer and instrument. Load the operating software called Thermospec by using the windows icon under Thermospec window or by typing "STNRUN" at the C: drive prompt in DOS.

8.5.2.2 Ignite plasma. Engage pump tubings. Under the menu heading SET UP, select CONTROL PANEL; then press F1 for Start Up followed by F9 for continue to begin the start up sequence which takes about 90 seconds.

8.5.2.3 Warm-up. Once the torch has been successfully lit, exit the start up submenu and go to the analysis menu. At the method prompt, enter "6010" and the peristaltic pump should begin turning and the levels adjusted to following:

Torch gas = HIGH
Auxiliary gas = LOW
Nebulizer gas = 0.588mL/min
Approximate RF Power (W) = 950
Pump rate (RPM) = 110

Fill the rinse water reservoir with the same matrix as the samples and set rinse time for 120 seconds. Fill the internal standard - ionization buffer reservoir.

8.5.3 Profile and prepare for sample analysis.

8.5.3.1 Place a 28ml autosampler cup filled with 5ppm As onto the last position (#19) on the "L" rack of the autosampler. Under the analysis submenu, press F6 to move autosampler and begin profile sequence. Once the autosampler has moved it will wait 35 seconds to allow for adequate uptake and equilibration of the test solution.

8.5.3.2 Start the profile sequence. Press F3 and then F1 to start the profile. The procedure takes approximately 63 seconds and returns a peak profile of the Arsenic line at 189.042 x 2nm (second order line).

8.5.3.3 Record the peak position and intensity in the daily maintenance logbook. The peak position should be within 0.1 units of the zero position. A drift greater than the specified tolerance could indicate a drastic barometric or thermal change since the last profile and warrants further investigation. (see trouble shooting.)

8.5.3.4 The calibration curve must consist of a blank and standards (refer to section 7.1.1). Use the average of two exposures for both standards and samples.

8.5.4 Prepare autosampler sequence.

Under Operation menu, select "Autosampler Setup". Load the default table name "trace" and enter the samples to be run under set 2. (maximum 192). Enter a CCV and CCB every 10 samples. Once finished, print out the table assignments by pressing F2.

8.5.5 Load autosampler with standards and samples according to the table printouts. A typical set-up should like this:

(set 1) Load autosampler L rack with 28mL cups

Position	Standard
5/45	ST00
5/46	ST01
5/47	ST02
5/48	ST03
1/1	CRI
5/42	PQL solution
5/44	ICV solution
5/43	ICB,CCB solution
1/2	ICSA solution
1/3	ICSAB solution
1/4	CRII solution
1/7 – 1/18	CCV solution (for long runs)
	CCB solution

(set 2) Load autosampler (48 position racks):

Position	Name
1	PBW (BATCH #)
2	LCSW (BATCH #)
3	SAMPLE
4	DUPLICATE D
5	SPIKE S
6	SERIAL DILUTION L
7	POST DIGESTION SPIKE
8	SAMPLE2
9	SAMPLE3
10	SAMPLE4
	CCV1
	CCB1
11 .. 20	10 more samples
	CCV1
	CCB1
21 .. 30	10 more samples
	CCV1
	CCB1
31 .. 40	10
	CCV1
	CCB1

41 .. 48,	8 more samples
rack 2, 1 .. 2	2 more samples
	CCV1
	CCB1

NOTE 1: The autosampler assigns the positions of the samples and QC in the order in which they are entered. Modifying an existing run by inserting samples may change the assignments of the samples.

NOTE 2: In order to take multiple uptakes from the same QC cup, the identical name must be entered. This would not allow for the numbering of CCV1/CCB1, CCV2/CCB2, etc. or the suffix of "I" or "F" on ICSA, ICSAB, PQL check solution used in CLP type packages.

8.6 Sample Analysis

8.6.1 Initiate autosampler run. Under the Operation menu, select Analysis, enter the method, press F9 for autosampler run; enter the desired autosampler table file and press F1, to start operation.

8.6.2 If the samples to be analyzed have been digested then all the calibration standards and quality control solutions used should be prepared using flame water.

8.6.3 Record the analysis sequence, the instrument identification, the date, the analyst's name, the analyst's signature, the time of analysis initiation, and the work order numbers on the bench sheet. Submit a copy of the bench sheet with the raw data.

8.7 Quality Control Requirements

8.7.1 IDL Study consisting of seven blanks run as samples must be performed every three months.

8.7.2 Calibration Curve

All analyses require that a calibration curve be prepared to cover the appropriate concentration range. The curve must have a correlation coefficient of 0.995. The calibration line is being generated using ordinary least squares. $y = mx + b$.

When multiple concentration standards are used, at least three calibration standards will be used. Alternatively, the initial calibration curve may be prepared daily with a minimum of a calibration blank and a single high standard. The resulting curve must then be verified with mid-level and low-level calibration verification standards. Acceptance range of +/- 20 % will be used for low-level calibration verification standard and +/- 10% for the mid-level calibration verification standard.

8.7.3 ICV/CCV

The ICV/CCV is run immediately after calibration. The CCV is run after every ten samples or every two hoursand the end of the analysis sequence. The ICV and CCV must be within 10% of the calibration with RSD < 5% from replicate integrations. When measurements for any element exceed the control limits the analysis is void for that element. The problem must be corrected and the samples reanalyzed.

Acceptance Criteria
ICV

Element	Control Limits (ppb)	True Value (ppb)
Aluminum	4500 - 5500	5000
Antimony	360 - 440	400
Arsenic	360 - 440	400
Barium	360 - 440	400
Beryllium	36 - 44	40
Boron	360 - 440	400
Cadmium	36 - 44	40
Calcium	4500 - 5500	5000
Chromium	360 - 440	400
Cobalt	360 - 440	400
Copper	360 - 440	400
Iron	4500 - 5500	5000
Lead	360 - 440	400
Magnesium	4500 - 5500	5000
Manganese	360 - 440	400
Molybdenum	360 - 440	400
Nickel	360 - 440	400
Potassium	4500 - 5500	5000
Selenium	360 - 440	400
Silicon	360 - 440	400
Silver	360 - 440	400
Sodium	9000 - 11000	10000
Strontium	360 - 440	400
Thallium	360 - 440	400
Tin	360 - 440	400
Titanium	360 - 440	400
Vanadium	360 - 440	400
Zinc	360 - 440	400

Acceptance Criteria
CCV

Element	Control Limits (ppb)	True Value (ppb)
Aluminum	45,000 – 55,000	50,000
Antimony	450 – 550	500
Arsenic	450 – 550	500
Barium	450-550	500
Beryllium	45 – 55	50
Boron	450 – 550	500
Cadmium	450 – 550	500
Calcium	90,000 – 110,000	100,000
Chromium	450 – 550	500
Cobalt	450 – 550	500
Copper	450 – 550	500
Iron	45,000 – 55,000	50,000
Lead	450 – 550	500
Magnesium	90,000 – 110,000	100,000
Manganese	450 – 550	500
Molybdenum	450 – 550	500
Nickel	450 – 550	500
Potassium	22,500 – 27,500	25,000
Selenium	450 – 550	500
Silicon	450 – 550	500
Silver	450 – 550	500
Sodium	90,000 – 110,000	100,000
Strontium	450 – 550	500
Thallium	450 – 550	500
Tin	450 – 550	500
Titanium	450 – 550	500
Vanadium	450 – 550	500
Zinc	450 – 550	500

8.7.4 ICB/CCB

The ICB and CCB are blank solutions. The ICB must be run immediately after the ICV. A CCB must be run immediately after each CCV. The absolute value of the ICB and CCB measurements should be less than or equal to the PQL.

For DOD projects, the value of the ICB and CCB measurements should be less than one half of the PQL.

For DOE projects, the value of the ICB and CCB measurements should be less than 3 x IDL.

Element	PQL (ppb)
Aluminum	200
Antimony	20
Arsenic	20
Barium	5
Beryllium	2
Boron	15
Cadmium	6
Calcium	1000
Chromium	5
Cobalt	5
Copper	10
Iron	150
Lead	10
Magnesium	250
Manganese	5
Molybdenum	5
Nickel	10
Potassium	250
Selenium	20
Silicon	50
Silver	3
Sodium	2500
Strontium	5
Thallium	30
Tin	25
Titanium	25
Vanadium	10
Zinc	20

8.7.5 ICSA/ICSAB

The ICSA and ICSAB must be run at the beginning and end of each analysis run or at a minimum of twice per eight hour shift. ICSAB must be run immediately following ICSA. ICSA contains interferents. ICSAB contains analytes plus interferents. The ICSA analytes results must be within absolute value of their reporting limits. If any element is outside this limit, then the analysis is void for that element. The ICSAB measurements must be within 20% of the true values. If any element is outside this limit, then the analysis is void for that element. The problem must be corrected and the element should be reanalyzed.

For DOD projects, the value of the ICSA measurements should be less than one half of the PQL.

Acceptance Criteria
ICSA

Element	Control Limits (ppb)	True Value (ppb)
Aluminum	400000 – 600000	500000
Calcium	400000 – 600000	500000
Iron	160000 – 240000	200000
Magnesium	400000 – 600000	500000

Acceptance Criteria
ICSAB

Element	Control Limits (ppb)	True Value (ppb)
Aluminum	400000 – 600000	500000
Antimony	480 – 720	600
Arsenic	80 – 120	100
Barium	400 – 600	500
Beryllium	400 – 600	500
Boron	800 – 1200	1000
Cadmium	800 – 1200	1000
Calcium	400000 – 600000	500000
Chromium	400 – 600	500
Cobalt	400 – 600	500
Copper	400 – 600	500
Iron	160000 – 240000	200000
Lead	40 - 60	50
Magnesium	400000 – 600000	500000
Manganese	400 – 600	500
Molybdenum	800 – 1200	1000
Nickel	800 – 1200	1000
Potassium	3500 – 6500	5000
Selenium	40 - 60	50
Silver	160 – 240	200
Sodium	4000 – 6000	5000
Strontium	800 – 1200	1000
Thallium	80 – 120	100
Tin	800 – 1200	1000
Titanium	800 – 1200	1000
Vanadium	400 – 600	500
Zinc	800 – 1200	1000

8.7.6 Linear Dynamic Range (LDR)

The LDR (Linear Dynamic Range) is to verify linearity of each element. The LR must be run at least once during analysis. Any element results

above the LR must be diluted and reanalyzed. Specific acceptance range ± 10% of the true value will be used. The LR will be verified with three standards every three months.

8.7.7 PQL (LOWCHECK)

The PQL (LOWCHECK) is to verify linearity at the reporting limit. The PQL (LOWCHECK) is prepared at approximately 5 times the MDL. The PQL must be run at least once during analysis and the acceptance range of ± 30% will be used. QSM clients, the acceptance range of ± 20% will be used.

8.7.8 BLK

The BLK is laboratory digested blank. If any element concentration in the digested blank is above the PQL, then all the samples associated with that blank which have concentrations greater than the PQL and less than ten times the blank concentration must be re-digested and reanalyzed for that element. If any element concentration in the digested blank is less than the negative of the PQL, then all samples associated with that blank must be reanalyzed.

Prep. Blank criteria for DOD projects must meet QSM requirements:
Any samples associated with a Prep. Blank that fail following criteria shall be re-digested and re-analyzed, except when the sample analysis results in a non-detect.

- if one-half the PQL is exceeded, and the concentration exceeds 1/10 of the measured concentration of any sample in the associated preparation batch or
- If the BLK concentration is greater than the 1/10 of the specified regulatory limit.
- If the concentration of common laboratory contaminants exceed the PQL

NOTE : If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifier.

8.7.9 BKS

The BKS are digested control samples. The BKS measurements must be within control limits. If any element concentration in the BKS is outside the control limits, then all the samples associated with that BKS must be re-digested and reanalyzed. The same spiking levels for the BKS are used for the MS.

8.7.10 Duplicates

One duplicate must be analyzed for each matrix type in each group of samples. If an element concentration is greater than or equal to five times the PQL, then the % RPD should be 20%.

$$\%RPD = \frac{\text{sample - dup}}{\frac{\text{sample + dup}}{2}} \times 100$$

If the duplicate falls outside the criteria, it must be noted on the raw data and written in the case narrative.

8.7.11 Matrix Spike and Matrix Spike Duplicate

The MS or MS/MSD sample analyses are designed to provide information regarding the digestion and methodology used for analysis. One matrix spike or MS/MSD samples are prepared for each matrix type in each batch of 20 samples. If the spike recovery for an element is outside ± 20% (for QSM clients, and if the sample concentration corresponding to the spiked sample is less than four times the spike added, then it should be noted in the case narrative and in the raw data. The acceptance limits for MS/MSD is ± 20% RPD.) For purposes of calculating the % spike recovery, sample results less than the instrument detection limit/reporting limit, should be assumed to be zero.

	Spiking Levels	
Element	Aqueous (ppb)	Solid (ppb)
Aluminum	5000	10000
Antimony	50	100
Arsenic	50	100
Barium	500	1000
Beryllium	25	50
Boron	500	1000
Cadmium	50	100
Calcium	5000	10000
Chromium	250	500
Cobalt	250	500
Copper	250	500
Iron	5000	10000
Lead	500	1000
Magnesium	5000	10000
Manganese	500	1000
Molybdenum	250	500
Nickel	250	500
Potassium	5000	10000
Selenium	50	100

Silicon	5000	10000
Silver	50	100
Sodium	5000	10000
Strontium	500	1000
Thallium	50	100
Tin	250	500
Titanium	1000	2000
Vanadium	250	500
Zinc	500	1000

NOTE: The DUP/SPIKE or MS/MSD requirements are subject to change to suit various client requirements.

8.7.12 Post Digestion Spike

A post digestion spike is analyzed for each batch of digested samples. The post spike concentration should be at 10-100 times the MDL. The post digestion spike can be analyzed on a diluted sample if the sample requires dilution. The criterion for post digestion spike is 75-125%. The criterion is not applicable when the spike addition is insignificant. (i.e. sample concentration is greater than 4x of the spike addition)

8.7.13 Serial Dilution

Transfer 2mLs of sample to 8mLs of flame water. One serial dilution is prepared for one sample of each matrix type in each group of samples. If the element concentration is fifty times the reporting limit or greater, then the % difference between the serial dilution and the sample should be 10%.

$$\% \text{ Difference} = \frac{\text{sample - dilution}}{\text{sample}} \times 100$$

8.7.14 Method of Standard Addition (MSA)

The MSA will be used if interference is suspected. When the method of standard additions is used, standards are added at one or more levels to portions of a prepared sample. This technique compensates for enhancement or depression of an analyte signal by a matrix.

8.7.15 Internal Standard

The internal standard should be approximately 40ppm. When internal standard is outside control limits of 50% the analysis will be repeated. The problem must be corrected and all samples reanalyzed.

8.7.16 Interelement Correction factor determination (IEC)

OVERVIEW:

Inter-element interference's are false spectral signals arising from other elements in the sample besides the analyte. By measuring the apparent false signal of interfering elements at known concentrations, corrections factors can be determined and applied to unknown samples. These are determined every six months or when routine interference check samples reveal the need for further adjustments.

PROCEDURE:

Set up, profile, and calibrate the ICP. Introduce clean single element standards at the linear range concentrations. Determine the correction factor using the following equation:

$$\text{(eq. 1)} \quad k_i = \frac{\text{apparent concentration of analyte}}{\text{known concentration of interferent}}$$

IEC's are then verified by introducing solutions of the interferences to determine the effectiveness of the IECs at canceling the apparent concentrations. IEC's are entered into the software controlling the ICP for application on unknown samples.

REPORTING:

After all inter-element correction factors have been determined IEC's are entered onto CLP forms 11A and 11B.

8.7.17 Hardness by Calculation

The preferred method for determining hardness is to calculate the results of separate determinations of calcium and magnesium.

Hardness, mg equivalent $CaCO_3$/L = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]

8.8 Instrument Shut Down

8.8.1 Aspirate flame water for several minutes.

8.8.2 Under Setup menu, select F7 (shutdown). This shuts off the torch and pump windings, goes through a cool down period of 90 seconds before shutting off the water recirculator and gas flows.

8.8.3 After the peristaltic pump stops, disengage the top two cartridges, leaving the tension setting untouched. The bottom cartridge supplies tension to the rinse reservoir and should be kept on to prevent back flowing of the rinse water.

8.8.4 Leave the circuit breaker on the RF power unit behind the instrument on at all times.

8.8.5 Leave the nitrogen purge gas on at all times.

8.9 Trouble shooting and corrective action

8.9.1 Problem: Stable plasma will not start.

Action: Make sure the pump tubing is clamped down and that there are no leaks in the tubing or spray chamber. Make sure the drain line is submerged under 6 inches of water in the drain waste vessel. Press reset on instrument, and there is

8.9.2 Problem: Profile peak position greater than 0.3 units from zero position.

Action: ICP may not have reached purge or thermal equilibrium. Check the nitrogen purge gas flow, replace nitrogen dewar if empty or low pressure prevents sufficient purging. If purge is sufficient, try re-profiling and recalculate spectrum shifter position. Set new vernier position and verify profile. Record new vernier position in daily maintenance log.

8.9.3 Problem: ICV fails for an element.

Action: (a) The instrument will need to be re-calibrated. (b) The profile may have drifted beyond 0.3 units from zero position. (c) The sample introduction system may have deteriorated since calibration indicating the nebulizer tip, pump windings, etc. may need cleaning or replacement. (d) The internal standard may have run out. (e) the ICV solution or sample introduction system may have been contaminated, perform additional rinse, refill ICV/CCV, and rerun to verify. (f) The calibration standards may need to be remade. (g) IEC's have changed.

8.9.4 Problem: ICB fails for an element.

Action: (a) The instrument will need to be re-calibrated. (b) The profile may have drifted beyond 0.3 units from zero position. (c) The sample introduction system may have deteriorated since calibration indicating the nebulizer tip, pump windings, etc. may need cleaning or replacement. (d) The internal standard may have run out. (e) the ICB solution or sample introduction system may have been contaminated, perform additional rinse, refill ICB/CCB, and rerun to verify. (f) The calibration standards may need to be remade. (g) The torch is dirty and will need to be cleaned.

8.9.5 Problem: ICSA or ICSAB fails for an element.

Action: (a) The spectrum shifter may need to be re-profiled, (b) the Interelement correction files may need to be changed or (c) new background points may need to be selected and new IEC performed.

9.0 SAFETY

9.1 Equipment

- Lab coat
- Safety glasses
- Gloves

9.2 Potential hazards

9.2.1 All samples and solutions are maintained 5% HCl and may contain high concentrations of metals a well. Safety glasses should be worn to protect the eyes from acid splashes. Gloves and lab coats should be worn to protect the hands and skin from spills or splatter. If any solution comes in contact with the skin, wash the area immediately with plenty of water and notify a supervisor. If any solution is splashed in the eyes, flush immediately and thoroughly in an eyewash basin and contact a supervisor immediately.

9.2.2 The ICP uses high voltage electricity and generates an RF field, so there is a potential risk of electrocution if shielding is circumvented. The TJA ICP has numerous safety interlocks to shut off power to the RF coils if there is a break in the shielding around the coils, if the supply of cooling water is lost, or if there is a loss of argon pressure.

10.0 WASTE MANAGEMENT

10.1 After analysis sample digestates must be held for six months, after which they are disposed of in accordance with the Hazardous Waste Disposal Procedure. For procedure and methods used for disposal refer to Standard Operating Procedures D.1 and D.2.

10.2 All other non-hazardous solutions may be washed down the drain with copious amounts of water.

11.0 POLLUTION PREVENTION

11.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

12.0 REPORTING REQUIREMENTS

12.1 The ICP run log must be filled out for each day's operations. The method, date & time of start and end of each run, standard sequence and sources, QC sequence and sources, and sequence of samples analyzed and date for preparation batch. In addition, each run must be recorded according to work orders, fraction numbers, corresponding elements completed, and analyst's initials.

12.2 The results reported on the raw data are in units of ug/L. To convert raw results of soil (solid) samples, multiply by the samples final volume (1 L) divided by the weight in grams. Then correct the results for solids if required. The final munits are mg/kg.

Example for solid sample

$$\frac{1 \text{ ug/L raw result X .1L}}{1.0 \text{ g sample}} = 0.1 \frac{\text{ug}}{\text{g}} = \frac{\text{mg}}{\text{Kg}}$$

12.3 If any maintenance is performed, routine or non-routine, the Maintenance log for the affected ICP shall be filled out if service people conduct maintenance on the instrument. The field service report is filed in a binder.

12.4 All solutions made must be entered in Solutions manager program and a print out is kept in a notebook in the laboratory.

12.5 Include copy of digestion log with weights and final volume for reviewer.

12.6 Include short narrative on why the run was needed, what problems were encountered, what actions were taken to correct them, and any future actions that will be needed.

13.0 METHOD PERFORMANCE

13.1 Per digestion methods MDL limits are obtained by digestion seven spiked replicates the same way as samples and analyzing them. MDL is defined as the minimum concentrating of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum.

14.0 METHOD DETECTION LIMIT

14.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

15.0 DEFINITIONS

15.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

SOP No: H.12

Title: Cold Vapor Analysis for Mercury in Accordance with SW846 Methods 7470A and 7471A.

1.0 SCOPE AND APPLICATION

1.1 The method detailed in this procedure explains the analysis of mercury in water, soil, sediment and TCLP extracts, by the Manual Cold Vapor Technique in accordance with SW846 methods 7470A and 7471A.

2.0 METHOD SUMMARY

2.1 To quantify both organic and inorganic forms of mercury in water, TCLP extracts, soil and sediment. The sample holding time is 28 days from sample collection. Sample digests containing mercury concentrations greater than the highest calibration standard will be diluted.

3.0 SAFETY

3.1 Gloves, safety glasses, and lab coat must be worn when performing any aspect of the mercury digestion or analysis process.

3.2 Mercury vapor is toxic. Caution should be taken during all phases of the digestion and analytical process. Care should be exercised so that mercury has no chance to be absorbed through the skin or inhaled.

3.3 The acids used to digest mercury are used full strength. Extreme care should be taken so that acid does not spill or splash. Transport samples and empty bottles on one of the 3 high concentration room spill carts. Transport acid using the 4 liter plastic carrying buckets.

3.4 Additional safety equipment includes mercury sponges, acid neutralization media, apron, a mercury scrubber, and face shield.

4.0 INTERFERENCES

4.1 Contaminants in the solvents, reagents, glassware and other sample processing hardware. These contaminants lead to discrete artifacts or to elevate baseline in gas chromatograms. All of these materials routinely must be demonstrated by running laboratory method blanks. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of the interferences will vary from source to source.

5.0 EQUIPMENT AND SUPPLIES

5.1 Mercury Digestion Equipment

- 300mL BOD bottles with glass stoppers
- hot plate
- hot water bath
- VWR Calibra M23
- VWR Calibra M27
- 100mL graduated cylinders
- 100mL volumetric flask (Class A)
- 200mL volumetric flask (Class A)
- balance capable of accurately measuring 0.200 grams (± .001g)
- squirt bottle filled with deionized water
- Parafilm squares for mixing standards in volumetric flasks
- thermometer traceable to NIST, alcohol based.
- Bottle top dispensers
- Fisher brand Finnpipette M9
- three 125mL plastic bottles with caps for ICV sub, ICV and Hg sub
- one 250mL plastic bottle with cap fo Hg SPIKE

5.2 Leeman Labs Automated Mercury Analyzer, Model HYDRA AA

5.3 Air source capable of delivering 1 liter of air per minute, argon source.

6.0 REAGENTS AND STANDARDS

- Concentrated Nitric Acid, Trace Metals Grade.
- Concentrated Sulfuric Acid, Trace Metals Grade.
- Concentrated Hydrochloric Acid, Trace metals Grade.
- Potassium Permanganate solution, 5% w/v. Dissolve 200g Potassium Permanganate to a final volume of 4L of deionized water.
- Potassium Persulfate solution, 5% w/v. Dissolve 100g Potassium Persulfate to a final volume of 2L of deionized water.

- Stock Mercury solution, 100µg/mL, traceable to NBS standard reference materials. (Source: Absolute Standards, Hamden, CT).

- Stannous Chloride (10%). Dissolve 200g of Stannous Chloride to 2L of 10% HCl. [Leeman requires the use of 10% Stannous Chloride in 10% HCl, DO NOT SUBSTITUTE Stannous Sulfate or Sulfuric Acid as described in the EPA method.]

- 10% Hydrochloric Acid. To a 20L HDPE carboy, add 10L deionized water, then add 2000mL of concentrated Hydrochloric Acid. Add deionized water to 20L final volume, cap container and mix. Record initials, date of preparation, and lot number of acid on side of carboy.

- Deionized water ASTM Type II or equivalent.

- Sodium chloride - Hydroxylamine Hydrochloride solution - Dissolve 240g of Sodium Chloride and 240g of Hydroxylamine Hydrochloride in deionized water and dilute to 2L (Hydroxylamine Sulfate may be used in place of Hydroxylamine Hydrochloride).

- ICV Solution(second source), 100µg/mL, traceable to NBS standard reference materials (Source: CPI International)

- 50% Aqua Regia digestion solution. To a 2.5 L Glass bottle, add 750mL DI water. Slowly add 750mL of concentrated Hydrochloric Acid, then add 250mL of DI water, followed by 250mL of concentrated Nitric Acid and mix well. Pour 50% Aqua Regia into plastic side bottle with bottle-top dispenser for use.

7.0 SAMPLE COLLECTION AND PRESERVATION

7.1 The holding time for mercury is 28 days.

7.2 Samples may be collected in either plastic or glass containers for aqueous samples, at least 500mL should be collected. For soil/solid samples, at least 5grams should be collected.

7.3 All samples should be stored at 4°C $\pm$ 2°C.

8.0 CALIBRATION AND STANDARDIZATION

8.1 2.0ppm Hg intermediate stock in 2% HNO_3 - Pipette 2.0mL of 100ppm Hg stock standard into a 100mL volumetric flask. Add 2% HNO_3 to a final volume of 100mL.

8.2 0.1ppm (100ppb) Hg working solution in 2% HNO_3 - Pipette 10mL of 2.0ppm Hg intermediate stock standard into a 200mL volumetric flask. Add 2% HNO_3 to final volume of 200mL.

8.3 1.0ppm Hg ICV intermediate solution in 2%HNO_3 - Pipette 1.0mL of 100ppm Hg stock standard into a 100mL volumetric flask. Add 2% HNO_3 to a final volume of 100mL.

0.1ppm Hg ICV working solution in 2% HNO_3 (see 8.5 and 8.10)

Standards for Water Samples and sample QC.

8.4 First add 100mls of DI water to six BOD bottles. Then with a pipette remove the appropriate amount of DI water then replace with equal amount of 0.1ppm working standard directly into the BOD bottle using the following volumes 0, 0.2, 0.5, 1.0, 5.0 and 10.0mL for calibration. The 0 standard is used for calibration, the ICB and CCB. The 0.2ppb standard is used for calibration and the reporting limit. The 5.0ppb standard is used for calibration and the continuing calibration verification standard. All other for mentioned standards are used for calibration only.

8.5 ICV (3.0ppb) – To BOD bottle, add 100mL of DI water using a graduated cylinder. Using a Pipette remove 3.0mL of DI water then add 3.0mL of Hg ICV working solution (0.1 ppm) into BOD bottle.

8.6 Preparation Blank (BLK) - Add 100mL DI water using graduated cylinder into BOD bottle.

8.7 Laboratory Control Sample (BKS) - Add 100mL DI water using graduated cylinder to BOD bottle. Using a pipette remove 1.0mL of DI water then Spike with 1.0mL of 0.1ppm working standard.

8.8 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) (1.0ppb) - For both MS and MSD samples, add 100mL of the selected QC sample using a graduated cylinder to a BOD bottle. Using a pipette, remove 1.0mL of sample. Next, Spike the sample with 1.0mL of 0.1ppm working standard. For TCLP samples, remove 11.0 mL of DI water, then add 10mL of sample, and then 1.0mL of 0.1ppm working standard.

Standards for Soil Samples and sample QC.

8.9 First add 10mL of DI water to four BOD bottles then remove with a pipette the following volumes of DI water 0, 0.2, 0.5 and 1.0 (ml), then replace with equal amounts of 0.1ppm working standard. For 5.0 standard add 5mL DI water then 5mL of 0.1ppm standard. For standard 10.0 add 10mL of working standard only. The 0 standard is used for calibration, the ICB and CCB. The 0.2ppb standard is used for calibration and the reporting limit. The 5.0ppb standard is used for calibration and the continuing calibration verification standard. All other for mentioned standards are used for calibration only.

8.10 ICV (3.0ppb) – To a BOD bottle pipette 3.0mL of 0.1ppm ICV working solution and 7.0mL of DI water.

8.11 Preparation Blank -(BLK) Pipette 10.0mL of DI water.

8.12 Laboratory Control Sample (BKS) - Pipette 7.0mls of DI water and Spike it with 3.0mL of 0.1ppm working standard.

8.13 Matrix spike (MS) and matrix spike duplicate (MSD) (3.0ppb)-For both MS and MSD samples, pipette 7mL of DI water to the BOD bottle. Using a pipette Spike the sample with 3.0mL of 0.1ppm working standard.

9.0 METHOD DETECTION LIMIT

9.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

10.0 METHOD PERFORMANCE

10.1 The MDL concentrations listed in the GPL MDL table are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

11.0 PROCEDURE (See Figure 1 for flow chart)

11.1 Preliminary Preparations.

11.1.1 Read workorders and test sheets for assigned samples, noting which samples are selected for duplicate and spike analysis. Review special instructions and submit sample request form to sample receiving for assigned samples.

11.1.2 Obtain samples from sample receiving and verify the identity of each sample. Record in the Mercury Digestion Log Form the date of digestion, analyst initials, and make an entry for each sample assigned (workorder-sample-fraction) using one line for each sample, standard, quality control check standard, blank, duplicate, spiked sample, and laboratory control sample.

11.1.3 Prepare BOD bottles for each entry by writing with permanent ink on the outside of the bottle the workorder number, the sample number, and fraction, using "MD", "MS" and "MSD" for Matrix Duplicate, Matrix Spike and Matrix Spike Duplicate.

11.2 Sample Digestion for Waters/TCLP Extracts and Water Standards

11.2.1 In the metals digestion lab measure 100.0mL of water or leachate sample into a clean, 100mL graduated cylinder and transfer sample into a clean, labeled 300mL BOD bottle. Stopper the BOD bottle, replace onto spill cart. For TCLP samples, measure out 90mL of DI water in a clean graduated cylinder and transfer that into a clean BOD bottle. Next, shake the closed TCLP bottle, and then pipette 10mL of the TCLP sample into the BOD bottle. Stopper the BOD bottles which hold the TCLP samples as well.

11.2.2 In the hood, to each bottle, add 5.0mL of concentrated Sulfuric Acid, 2.5mL of concentrated Nitric Acid, and 15mL of 5% Potassium Permanganate solution. Mix sample after each addition. After the first 3

reagents have been added let the samples sit for 15 minutes. If a sample does not retain a purple color after sitting for 15 minutes, add additional Potassium Permanganate in 15mL increments until the sample is able to sit for 15 minutes without losing color. Do not add more than 32mL of Potassium Permanganate to any sample. Record the final amount of permanganate added into the digestion log.

Note: If additional permanganate is added, the final volume must be changed accordingly on the bench sheet for correct final calculation of results.

11.2.3 Add 8mL of 5% Potassium Persulfate solution to each sample mix.

11.2.4 Preheat water bath to 60°C by changing the set point on the temperature controller to 60°C. This should be done while samples are being measured out. Add the correct amount of water for the day's digestion before hand. Make sure to cover the water bath with the heat retention plastic balls. Once all required reagents are added, place the BOD bottles gently into the water bath. Depending upon how low the temperature drops by the addition of the BOD bottles depends upon what the new set point should be changed to in order to heat the bottles to the 93°C-98°C range. The analyst should make these judgment calls each day for each digestion. Once the water bath is within the desired temperature range, change the set point to 95°C. Make sure that the temperature probe is not touching the bottom of the water bath, yet is still below the water line for an accurate reading. Temperature should be checked with a NIST traceable thermometer. Heat at temperature for 2 hours.

11.2.5 Remove samples from water bath and allow samples to cool to room temperature. Add 6mL of Sodium Chloride Hydroxylamine Hydrochloride solution and swirl bottle to mix so that all traces of permanganate will be reduced. [Caution: the addition of this solution produces an effervescence which could cause back-pressure build up and force out the glass stopper. After adding the hydroxylamine to the sample, let the pressure vent off by removing the glass stopper periodically.]

11.2.6 Samples are ready for analysis.

11.3 Sample Digestion for Soils/Sediments and Soil Standards

11.3.1 Use an analytical balance to measure a representative 0.600-1.00g, or three separate scoops of approximately 0.2 grams each of soil, or sludge, Laboratory Control Sample, solid or sediment sample into a 300ml BOD bottle. Record measurement to 3 significant figures. Stopper the BOD bottle. When all samples have been measured, return the unused portion of sample to sample receiving area. Set MS and MSD bottles aside for spiking.

11.3.2 In the hood, add 10mL 50% Aqua Regia digestion solution to each BOD bottle. Add 10mL of distilled water to each BOD bottle in order to rinse any remaining sample residue and to cover the entire sample. Preheat water

bath to 60°C by changing the set point on the temperature controller to 60°C. This should be done while samples are being measured out. Add the correct amount of water for the day's digestion before hand. Make sure to cover the water bath with the heat retention plastic balls. When Samples are ready to have reagents added to them, change the set point to 86°C. Once all required reagents are added, place the BOD bottles gently into the water bath. Depending upon how low the temperature drops by the addition of the BOD bottles depends upon what the new set point should be changed to in order to heat the bottles to the 93°C-98°C range. The analyst should make these judgment calls each day for each digestion. Once the water bath is within the desired temperature range, change the set point to 95°C. Make sure that the temperature probe is not touching the bottom of the water bath, yet is still below the water line for an accurate reading. Temperature should be checked with a NIST traceable thermometer. Heat at temperature for 2 minutes.

11.3.3 Remove bottles from bath and allow to cool. Add 15mL of Potassium Permanganate then 40mL of distilled water to each bottle. Preheat water bath to 60°C by changing the set point on the temperature controller to 60°C. This should be done while samples are being measured out. Add the correct amount of water for the day's digestion before hand. Make sure to cover the water bath with the heat retention plastic balls. When Samples are ready to have reagents added to them, change the set point to 86°C. Once all required reagents are added, place the BOD bottles gently into the water bath. Depending upon how low the temperature drops by the addition of the BOD bottles depends upon what the new set point should be changed to in order to heat the bottles to the 93°C-98°C range. The analyst should make these judgment calls each day for each digestion. Once the water bath is within the desired temperature range, change the set point to 95°C. Make sure that the temperature probe is not touching the bottom of the water bath, yet is still below the water line for an accurate reading. Temperature should be checked with a NIST traceable thermometer. Heat at temperature for 30 minutes.

11.3.4 Remove bottles and allow samples to cool to room temperature. Add 50.0mL DI water. Add 6mL of Sodium Chloride Hydroxylamine Hydrochloride solution to reduce the excess permanganate.

11.4 Running, Tuning and Calibration of Instrument HYDRA AA

Soil, tissue and thick non-aqueous liquid samples can share standards. Water, Extracts and thin Non-Aqueous Liquid samples can share standards. These two groups cannot share standards and must be analyzed separate from each other.

11.4.1 Maintenance Check: pump tubing (for worn down areas), reductant level, waste vessel (waste drain tubing should never be below the surface of the liquid waste), rinse level (rinse bottle should be filled before software is

started), dry the little piece of tubing leading from the Liquid/Gas Separator to the Dehydrator (use compressed nitrogen), look for any blockage in all of the tubing (usually found where one piece of tubing connects with another), make sure that the printer has enough paper for the run

11.4.2 Double click on the "WinHg" icon on the desktop, this will open th WinHg Runner. Select the "Protocol" and "Dataset" to be used for the run. Click on the "Database" icon and also the "Rack Editor" icon.

11.4.3 In the WinHg Database, click on the "Cal Curve" tab. The click on the "Calib Coeffs" and "New Cal" buttons to clear the graph and allow for a new set of standards to be used for the run.

11.4.4 In the WinHg Rack Editor, enter cup identification information in both the "sample ID" and "extended ID" columns. In the fifth column, enter the codes for checking the ICV, ICB, CRA, CCV, and CCB. C2 for the ICV, C3 for the ICB, C4 for the CRA, C5 for the CCV, and C6 for the CCB. In the row for the first cup in the fifth column, place CP after the check codes. (Example: C2 C3 C4 C5 C6 CP) Every 10th sample should have a CCV and CCB action code in its fifth column. (Example: C5 C6) The fifth column for the last sample should also contain the action codes for the CCV and the CCB. If a sample is diluted by any amount, make a mark in the extended ID column attached to the end of the extended ID. (Example: 01-001-1/1:10 for a times 10 dilution.) Save the rack as the year first, then month, then date. (Example: 051117 for November 17th, 2005) If there is more than one rack prepared in a day, save them with letters descending from A. (Example: 051117A and 051117B for racks saved after the first one on November 17th, 2005)

11.4.5 Click on the WinHg Runner window. Click on the "Main" tab. Under the Printer Control panel, check to make sure that the Next Page number is at 1 and that the Lines Waiting number is at 0. If there are no Lines Waiting and the Next Page number is not 1, click on the "Reset" button next to it. If there are Lines Waiting, click on the "Form feed" button. This information might be needed for a past run. If this is the case, click on the "Reset" button once it in done printing. Lines Per Page should be set no higher than 63 and no less than 50.

11.4.6 Click on the "Standard" tab. Click the buttons for the standards and triplicate repetitions. S1 for the 0.0 standard, S2 for the 0.2 standard, S3 for the 0.5 standard, S4 for the 1.0 standard, S5 for the 5.0 standard, S6 for the 10.0 standard. Rep1, Rep2 and Rep3 for repetition 1, 2 and 3. Load the standards rack into the correct slot with all the standards in it.

11.4.7 Click on the "Sample" tab. Click on the "Start New Batch" button and click okay when prompted. No Batch ID is required. Then click on the WinHg Database window. Click on the "Report" tab. Make sure that only the new Batch is selected in the Batch List panel. Under the Record panel, there should only be a number under the "Seq" column and under the "Rec" column next to it should be BAT.

11.4.8 Return to the WinHg Runner window. In the Autosampler Run panel, select the correct Rack to be used (Example: 051117), then which samples in that Rack by selecting the first cup in the Rack to be tested under Start Cup (Example: 1) and the last cup in the Rack to be tested under End Cup (Example: 25). The Cups Per Rack should be 44.

11.4.9 Click on the "Control" tab. Click on in the Hg Lamp panel to start warming up the instrument's mercury lamp. After the lamp has warmed up sufficiently, attach rinse and reductant tubing to appropriate bottles and put the pump cassettes on. Turn on both the Gas and Pump by clicking the appropriate "On" buttons. Once the liquids have passed through the instrument for about one minute, click the "Adjust" button. This will take about two minute to complete. When prompted, click "Okay" and then click on the "Utility" tab. Under the Action menu, select DAQ Test and then click the "Do Action" button. Record the reading it gives under Results for the Sample and the Hg Lamp Volt. The Sample reading should be between 700,000 and 1,720,000. The Hg Lamp Volt reading should be between 5.000 and 9.500 V.

11.4.10 Next, go back to the "Control" tab and once again turn "On" the Gas and Pump. Next, move the tubing from the small 10% HCl bottle and place it into the 10% Stannous Chloride bottle. Let the instrument run like this for no less than 1 minute and no more than 5 minutes.

11.4.11 Click on the "Standard" tab. Click on the "Stnd Auto" button. This will run standards 1-6 in triplicate if you have prepared properly. Make sure that the WinHg Database is on the "Cal Cruve" tab screen. The allowed %RSD for the 2nd standard is 20%. The allowed %RSD for the 3rd standard is 10%. Standards 4 through 6 have a %RSD maximum of 5%. If Rho is greater than 0.998000, the deviation of the blank standard is within 0.1 ug/L and the other standards are within 5% of their true values, click the "Accept" button and then the "Print" icon. The 2nd standard is exempt from the 5% variance. All of these requirements must be met for the curve to be acceptable to use.

11.4.12 Go back to the WinHg Runner window. Click on the "Sample" tab. Click on the "Run Auto" button.

11.4.13 When the run is over, turn off the Hg Lamp and move the tubing from the reductant bottle to the extra 10% HCl rinse bottle. If the last page has not been printed out yet, click on the "Form feed" button in the "Main" tab. Then click the "Reset" button after the last page has printed in the Printer Control panel. Click on the "Report" tab in the WinHg Database window. Click on the "Generate Report" button. Under Format, select PRN file. Click the "..." button for where to save the PRN file. Save the file on the T drive via the Metals/hg pathway. Save file as Hg and date. (Example: HG051117 for November 17th, 2005) If multiple files are needed in one day name them in the same method that multiple racks are saved in. Once the file is saved, click the "Generate" button. The "Total Lines Generated" should be one more than the number under "Included In Report" in the WinHg Database window.

11.4.14 Allow the Pump and Gas to run for several minutes to clean the instrument's tubing and sterilize it. After a reasonable amount of time, remove the tubing from the spare 10% HCl bottle and the Rinse bottle. Let the instrument draw in air for about 5 minutes, then turn off the Gas and Pump. Remove the pump cassettes. Close all windows to shut off program.

Trouble Shooting Guide. Refer to Leeman Labs Manual.

12.0 DATA ANALYSIS AND CALCULATION

12.1 The linear regression analysis is being used to generate the initial calculation line. The equation used in calculating the curve is: $y = mx + b$

12.2 The linear regression analysis is being used to generate the initial calculation line

12.3 The results reported on the raw data are in units of ug/L. To convert raw results of soil (solid) samples, multiply by the samples final volume (0.1L) divided by the weight in grams. Then correct the results of solids if required. The final units are mg/kg.

Example for solid sample:

$$\frac{1 \text{ ug/L raw result} \times 0.1\text{L}}{.6\text{g}} = 1.67 \frac{\text{ug}}{\text{g}} = \frac{\text{mg}}{\text{kg}}$$

12.4 In addition to the accurate reporting and handling of raw data, there are records that need to be maintained for internal auditing purposes.

12.4.1 Before analysis is completed, record on the raw data the date of analysis, the analyst, the Mercury standard's source, lot number, and GPL ID, the ICV's number, and GPL ID, the LCSS's number and GPL ID, a list of the cases included in the run, and the instrument upon which the analysis was performed. Note the source for the spike used in the matrix spike sample and % Recovery for the spike in the raw data.

12.4.2 Complete the Bench Sheet for Mercury with the following information: Instrument ID, Date of analysis, Analyst name and signature, Time analysis was initiated, case and SDG, work order numbers, sample sequence run, dilution factors and comments.

12.4.3 Complete the Instrument Use Log with the following information: Date, Method, Case, Analyst and Comments.

12.4.4 If maintenance or repair was required on the instrument, all relevant information must be recorded in the Maintenance Log. This Log will be maintained with all field service reports for each instrument.

13.0 QUALITY CONTROL

13.1 Any results that exceed the highest calibration standard are diluted and reanalyzed. (LR = highest calibration standard)

13.2 The mercury auto-analyzer is capable of detecting parts per trillion levels of mercury when optimized. However, the laboratory reports lowest standard concentration level.

13.3 Calibration standards must be prepared fresh daily from stock standards. The deviation of each standard must be within 5% of the standard's concentration or the run must be terminated and the instrument re-calibrated. The 5% criteria does not apply to calibration standard of the CRQL 0.2ppb. The correlation coefficient must be _ .995 or the run must be terminated and the instrument re-calibrated.

13.4 Initial Calibration Verification Check solution. An ICV solution is prepared from an independent standard as the calibration standards are prepared. This solution is then digested with the samples and is the 1st check standard to be run. If recovery is outside 90-110% window, the run terminates. Recalibrate and rerun the ICV and ICB.

13.5 Continuing Calibration Check solution. The middle standard is used as the CCV solution. Window of acceptance for CCV is 80-120%. The CCV is run once every 10 samples.

13.6 Initial and Continuing Blank Solution. A blank solution, separate from the calibration blank, is digested with the standards and analyzed at after each ICV and CCV. Criteria is absolute value of +/- 0.2ug/L. For DOD project, the criterion is 2 X MDL. The CCB is run after the CCV.

13.7 PQL solution. A solution of 0.2ug/L Hg is prepared as calibration standards are being prepared and is analyzed at the beginning of the run following the ICB.

13.8 Preparation Blanks are digested exactly as the samples. The same type of container, utilization of the same amount of reagents, and are assigned to the same digestion batch. The blanks are placed in sequence every twenty samples. Criterion is absolute value of +/- 0.2ug/L. For DOD project, the criterion is one half of the Reporting limit.

13.9 Laboratory Control sample. A blank solution is spiked at 1.0ug/L to check the spiking procedure and the digestion process. Acceptance criteria is 80 –120%.

13.10 Spike Sample. 0.6 – 1.0g of sample is spiked with 1.0mL of 0.1ppm Hg standard for water, 3.0mL for soil before addition of reagents or digestion. One spike is prepared every 20 samples of the same matrix. If the matrix spike is outside 75 -125% (80 – 120% for QSM clients) criteria, a post digestion spike must be analyzed (sec 13.11). It also must be noted in the case narrative.

13.11 Post digestion spike. This is performed if the matrix spike is outside the criteria. The post digestion spike should fall within the midrange of the calibration curve and

should be within 75-125%. If the post digestion spike is outside criteria and the sample result is above the reporting limit an MSA should be performed to quantify the result. The MSA should be used for all samples of similar matrix within the batch.

13.12 Duplicate. One duplicate every twenty samples will be performed. The duplicate is prepared before addition of any reagents or digestion.

14.0 POLLUTION PREVENTION

14.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

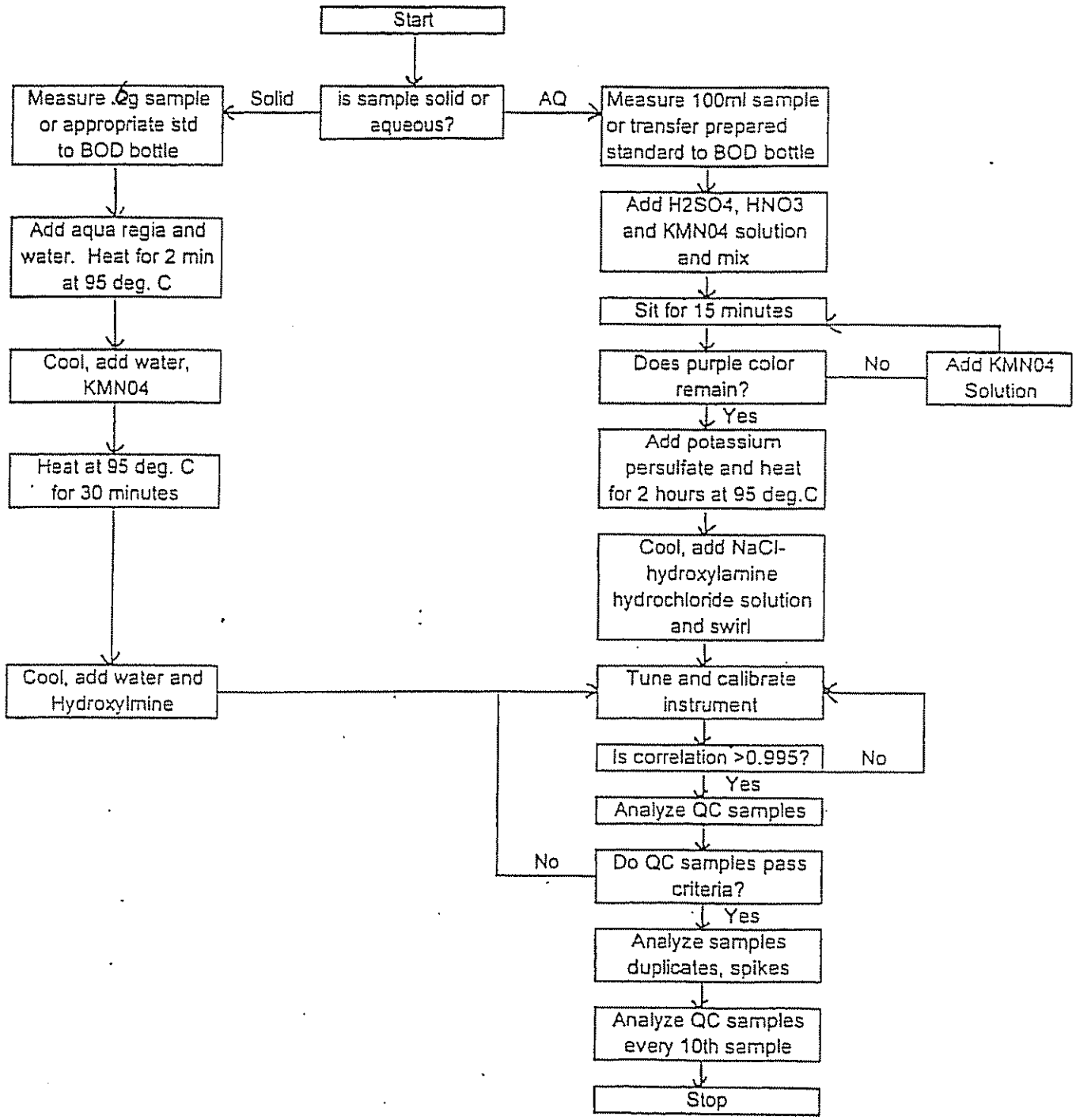
15.0 DEFINITIONS

15.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

16.0 REFERENCES

- SW846 method 7470A and 7471A.
- Leeman Labs HYDRA AA Manual, Leeman Labs, Inc., Lowell, MA

Figure 1
Procedure Flow Chart



SOP No: H.7

Title: Toxicity Characteristic Leaching Procedure (TCLP)

Scope: This procedure is designed to determine the mobility of organic and inorganic contaminants present in soil, sludge, oil, liquid-liquid suspension, and multiphasic wastes. Holding times are dictated by the analytical methods used to determine metallic and organic constituents.

1.0 PURPOSE

1.1 A representative sample of solid waste is leached with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. The liquid phase of the leachate is filtered from the solid phase by a 0.6 to 0.8um glass fiber filter. The filtered leachate is then analyzed to determine if any of the threshold levels for eight elements (arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver), four pesticides (Endrin, Lindane, Methoxychlor, Toxaphene), two herbicides (2,4,5-trichlorophenoxypropionic acid, 2,4-dichlorophenoxyacetic acid), and twenty five organics (Attachment 1) have been exceeded. If chemicals are present at or above the specified regulatory levels, the waste is a "TC waste", and is subject to all RCRA hazardous waste requirements.

2.0 REFERENCES

- Toxicity Characteristic Leaching Procedure (TCLP), Method 1311, Revision 0, July 1992

3.0 EQUIPMENT AND SUPPLIES

3.1 Rotary Agitation Apparatus:

An acceptable rotary agitation apparatus is one which is capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm. Suitable devices are available from Associated Design and Manufacturing Co. in Alexandria, VA, Lars Lande Manufacturing of Whitmore Lake, Michigan, IRA Machine Shop and Laboratory, Santurce, Puerto Rico, REXNARD in Milwaukee, Wisconsin and Analytical Testing and consulting services, Inc. of Warrington, Pennsylvania.

3.2 Extraction Vessel:

Zero-Headspace Extraction Vessel (ZHE).
This device is for use only when the waste is being tested for the mobility of volatile constituents (see Table 1). The ZHE is an extraction vessel that allows for liquid/solid separation within the device, and which effectively precludes headspace (as depicted in Figure 3). This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without having to open the vessel. Suitable ZHE devices are available from Associated Design and Manufacturing Company, Alexandria, Virginia, Millipore Corp., Bedford Massachusetts, and Analytical Testing and Consulting Services, Inc., Warrington, Pennsylvania.

Two liter polyethylene bottle for inorganic extractions.

Two liter teflon bottle or borosilicate glass for non-volatile organic extractions.

3.3 Filter Holder:

When the waste is being evaluated for other than volatile compounds, a filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation is used (50psi). Filter holders shall have a minimum internal volume of 300mL and be equipped to accommodate a minimum filter size of 47mm (Filter holders having an internal capacity of 1.5L or greater and equipped to accommodate a 142mm diameter filter are recommended). Vacuum filtration is only recommended for wastes with low solids content (<10%) and for highly granular (liquid-containing) wastes. All other types of wastes should be filtered using positive pressure filtration. Plastic filter holders may be used only when inorganic containers are of concern. Suitable filter holders are available from Nuclepore Corp., Pleasanton, California, Micro Filtration Systems, Dublin, California, and Millipore Corp., Bedford, Massachusetts.

3.4 Filters:

Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8um, or equivalent. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1.0N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1L per rinse is recommended). Glass fiber filters are fragile and should be handled with care. Suitable filters are available from Whatman Laboratory Products, Inc., Clifton, New Jersey.

3.5 pH meter

3.6 ZHE extract collection devices:

TEDLAR bags or glass, stainless steel or PTFE gas tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the

ZHE device. The devices listed are recommended for use under the following conditions.

If a waste contains an aqueous liquid phase or does not contain a significant amount of non-aqueous liquid (i.e., <1% of total waste), the Tedlar bag should be used to collect and combine the initial liquid and solid extract. The syringe is not recommended as its use causes unnecessary sample manipulations.

If a waste contains a significant amount of non-aqueous initial liquid phase (i.e., >1% of total waste), the syringe or the Tedlar bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

If the waste contains no initial liquid phase (is 100% solid) or has percent dry solids <0.5%, either the Tedlar bag or the syringe may be used.

3.6 ZHE extraction fluid transfer devices:

Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a constant displacement pump, a gas tight syringe, pressure filtration unit or another ZHE device).

3.7 Laboratory balance:

Any balance accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

3.9 Disposable weigh dishes

3.10 Spatula for mixing samples

3.11 Structural Integrity Tester

3.12 Magnetic plate/stir bar

3.13 Beaker, glass 150ml

3.14 Watch glass of appropriate diameter to cover beaker

3.15 1mm sieve

3.16 9.5mm sieve

4.0 REAGENTS

4.1 ASTM Type II water available from the Culligan water system

4.2 1.0 N Hydrochloric acid (HCl), made from ACS reagent grade.

4.3 1.0 N Nitric acid (HNO_3) made from ACS reagent grade.

4.4 5.0 N Sodium hydroxide (NaOH) made from ACS reagent grade.

4.5 Glacial acetic acid (HOAc) ACS reagent grade.

4.6 Extraction fluid:

Extraction fluid #1:
This fluid is made by adding 57mL glacial HOAc to 500mL of the appropriate water, adding 128.5mL of 5.0 N NaOH, and diluting to a volume of 10 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05. After the extraction fluid is prepared, the pH must be taken using a pH meter and recorded in the TCLP log book.

Note: Concentrated extraction fluid 1 may also be purchased from High Purity Standards, which is then diluted up to volume for a pH of 4.93 ± .05.

Extraction fluid #2:
This fluid is made by diluting 5.7mL glacial HOAc with ASTM Type II water to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05. Take the pH of the extraction fluid with a pH meter and record in the TCLP log book.

5.0 PROCEDURE

5.1 Sample collection and preparation:

5.1.1 At least two separate representative samples of a waste should be collected. If volatile organic are to be tested, a third sample should be collected. The first sample is used in several preliminary TCLP evaluations which determine the percent solids of the waste. This will ascertain if the waste contains insignificant solids and will be its own extract upon filtration, or if the waste requires particle-size reduction using the Structural Integrity Procedures. These preliminary evaluations will also determine which of the two extraction fluids are to be used for the non-volatile and inorganic TCLP extraction of the waste. The second and third, if necessary, samples will be used for the actual TCLP extraction for organics and inorganics.

5.1.2 Preservatives shall not be added to samples before TCLP extraction.

5.1.3 Samples can be refrigerated unless refrigeration results in irreversible physical changes to the waste such as precipitation.

5.1.4 When the waste is to be analyzed for volatiles, care should be taken to ensure the sample meets with no, or minimal, air contact. It is recommended that any particle-size reduction be conducted prior to or during sample collection.

5.1.5 TCLP extracts should be prepared and analyzed as soon as possible following extraction. If storage is required, the extracts shall be kept at 4°C. Samples for volatile analysis shall not be allowed to come into contact with air and shall be kept under zero-headspace conditions.

5.1.6 When analyzing TCLP extract for metals, the glass-fiber filters must be acid-washed prior to filtration. This is done by rinsing the filter with 1.0-N nitric acid followed by at least three rinses with type II water. Each rinse should take approximately one-liter of type II water per rinse. Glass-fiber filters are very fragile, handle with blunt forceps to avoid damage to filters.

5.2 Preliminary TCLP evaluations:

Preliminary TCLP evaluations are performed on a 100 gram representative sample of the waste in question. This subsample will not actually undergo TCLP extraction.

5.2.1 Percent solids:

Percent solids determination is only applicable to liquid and multiphasic samples. If the sample is a dry solid, (i.e. powder or soil), proceed to step 5.2.2. If the sample is a liquid or multiphasic, liquid/solid separation to make a rough determination of percent solids must be performed. This involves filtration using a positive pressure filtration device. The use of this device is as follows:

5.2.1.1 Pre-weigh the glass-fiber filter and the container that will receive the filtrate separately and record the initial weight of each.

5.2.1.2 Place the base plate on the tripod and position the filter support screen on top. Place the pre-weighed, acid-washed filter on the support screen. Do not pre-wet filter. Place the main body cylinder on top of the base plate and filter assembly and secure using thumbscrews provided.

5.2.1.3 Using a disposable weighing dish or other suitable container, weigh out a representative 100g of the waste. Allow to stand so the solid phase will settle.

5.2.1.4 Transfer the subsample into the filter holder, place top plate and pressure regulator on top and secure with the thumbscrews provided. Apply gentle pressure (10 to 15 psi) until all liquid passes through the filter. If this point is not reached under gentle pressure, increase the pressure slowly in 10psi increments to a maximum of 50psi. Filtration is complete when no more liquid is expressed or when pressurizing gas moves through the filter. The

material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Instantaneous application of high pressure may degrade the glass-fiber filter and may cause premature plugging or rupture. Some wastes, such as oily wastes and paints will contain some material that appears to be liquid and will not filter. If this is the case, the material within the filtration device is defined as a solid. The original filter is not to be replaced under any circumstances. Only one filter is to be used.

5.2.1.5 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate and container. The weight of the solid phase is determined by subtracting the weight of the liquid phase from the total weight of the sample before filtration. Record the weight of each phase. Calculate the percent solids as follows:

$$\% \text{ solids} = \frac{\text{Weight of solid}}{\text{Total weight of waste}} \times 100$$

If the sample appears to have solids greater than 0.5%, proceed to step 5.2.2.

5.2.1.6 If the sample appears to have <0.5% solids, determine the exact percent solids using the procedure below.

Dry the filters and residue at 100 ± 20°C until two consecutive weighings yield the same value within ± 1%. Record the final weight.

Calculate the percent solids using the following equation:

$$\frac{\text{Weight of filter and solid - weight of filter}}{\text{initial weight of waste sample}} \times 100 = \% \text{ dry solids}$$

If the sample has <0.5% solids, discard the solid and treat the liquid as the extract. Proceed to step 5.3.8.

5.2.1.7 The solid material obtained from the above procedure and all samples that do not contain filterable liquids must be evaluated for particle size. If the solid material has a particle size small enough to pass through a 9.5 mm standard teflon-coated sieve, proceed to next step. If the particle size is larger than specified above, prepare it by crushing, cutting, or grinding the sample until it will pass through a 9.5mm teflon-coated sieve. If the material is in a single piece, it must be subjected to the Structural Integrity

Procedure. Samples to be evaluated for volatile organics should already be at optimum particle size and should not undergo structural integrity testing or particle size reduction.

5.2.2 Determination of appropriate extraction fluid:

This step will be used only when the percent solids are >0.5% and the TCLP extraction is being performed for non-volatile or inorganic compounds. TCLP extraction for volatiles uses only extraction fluid #1. If extraction will not occur for non-volatiles and inorganics, proceed to step 5.4, TCLP Extraction Procedure for Volatile Organics (VOA).

5.2.2.1 Reduce the solid to a particle size of approximately 1mm in diameter or less and weigh a 5.0g subsample of the waste to a 500ml beaker.

5.2.2.2 Add 96.5ml of type II water, cover with a watch glass and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH with a pH meter. If the pH is <5.0, use extraction fluid #1 and proceed to step 5.3.

5.2.2.3 If the pH is >5.0, add 3.5ml 1.0-N HCl, swirl together, cover with a watch glass, and heat at 50°C for 10 minutes. Let cool to room temperature and measure the pH with a pH meter. If the pH is <5.0, use extraction fluid #1. If the pH is still >5.0, use extraction fluid #2. Proceed to step 5.3.

NOTE: The sample of waste used for preliminary evaluations shall not be used any further. The other samples of the waste shall be used for the actual extractions.

5.3 TCLP Extraction for Inorganics and Non-volatiles (BNA):

A minimum sample size of 100g is required for TCLP extraction. Enough solids should be generated by filtration to produce sufficient volume of TCLP extract required for all analyses. If the amount of extract generated by a single TCLP extraction is not sufficient to support all analyses required, a second extraction using a second 100g subsample of the same waste may be performed and the extracts from both be combined with any initial filtrates, if compatible, to form the final extract. This extract may then be aliquoted for analysis. Particle size reduction may be necessary. The solid portion of the sample must fit through a 9.5mm sieve. See section 5.2.1.7.

5.3.1 If the waste is obviously a dry solid which will yield no liquid, mix thoroughly and weigh out a minimum 100g subsample. Proceed to step 5.3.10.

5.3.2 If the sample is liquid or multiphasic, filtration is required for liquid/solid separation. This involves the positive pressure filtration device used in step 5.3.6.

5.3.3 Pre-weigh the container to receive the filtrate. Assemble the positive pressure filter holder. Be certain the filter has been acid-washed if the TCLP extract will be analyzed for metals (see step 5.1.6).

5.3.4 Weigh out a representative subsample (100g minimum) and record the weight. If the waste contains <0.5% solids, as shown by the preliminary tests, the waste will be its own extract after filtration. Enough sample should be filtered to ensure enough TCLP extract for all analyses. For wastes containing >0.5% solids, as shown by preliminary tests, use the percent solids information obtained to determine the optimum total sample size for filtration. Enough solid should be generated to support all analyses to be performed on the TCLP extract.

5.3.5 After weighing, the subsample should be allowed to stand to permit the solids to settle to aid in filtration.

5.3.6 Transfer the waste sample (both phases) into the filter holder, place the top plate and pressure regulator on top and secure into place. Gradually apply gentle pressure of no more than 10psi. If liquid is not expressed under gentle pressure, increase pressure in increments of 10psi, to a maximum of 50psi, or until pressurizing gas moves through the filter. If at this point, the pressurizing gas has not moved through the filter and no additional liquid has been expressed in a 2-minute interval, filtration is complete.

5.3.7 As in the preliminary steps, any material left in the filter holder is defined as the solid phase of the waste and the filtrate is defined as the liquid phase. The liquid phase may either be analyzed as a separate layer of the sample or it may be stored at 4°C for later combination with final extract for analysis.

5.3.8 If the waste was determined to have <0.5 % solids, the liquid filtrate is its own extract and may be analyzed at this time. If the waste was determined to have >0.5 % solids and particle size reduction was not needed, transfer the solid material into the extractor vessel (polyethylene for metals, teflon for organics) including the filter used to perform the separation. If the sample is a dry solid, weigh a 100-g minimum subsample and place in the appropriate extraction vessel. Proceed to step 5.3.10.

5.3.9 If the waste was determined to have >0.5 % solids and required particle size reduction, prepare the solid by crushing, grinding or cutting the waste until the optimum surface area has been obtained. Proceed as for solids described in step 5.3.8.

5.3.10 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\frac{20 \times \%\ \text{solids} \times \text{weight of waste filtered}}{100} = \text{weight of extraction fluid}$$

Slowly add this amount of extraction fluid, (type which was determined in 5.2.2) to the extraction vessel. Close the bottle tightly, place extraction vessel into a rotary agitator and extract for 18 ± 2 hours at 23° ± 2°C. Note the room temperature in the log book. Vent the bottle at 15, 30, and 60 minute intervals for the first hour of extraction.

After extraction, allow solution to stand to permit the solid phase to settle before filtration. Note the room temperature in the log book. If the room temperature is outside 23 ± 2°C, the samples must be re-extracted. Wastes that settle slowly may either be centrifuged or allowed to stand overnight to ensure good separation. During this time set up the positive pressure filter apparatus.

5.3.11 Wet the filter with a small portion of the liquid phase form the waste or from the extraction solution. Transfer the remaining solution into the filter apparatus and apply pressure as described in step 5.3.6 until all liquid passes through the filter. Stop filtration when the pressurizing gas moves through the membrane filter. If this point is not reached under gentle pressure, increase the pressure in 10psi increments to a maximum of 75psi. Halt filtration when liquid flow stops.

5.3.12 Any liquid saved from step 5.3.7 should now be combined with the final filtrate to form the TCLP extract. It is this mixture which shall be analyzed for all parameters of interest. If the sample was a dry solid, the final filtrate is the TCLP extract.

Note that if recombinations of the liquid phase to the TCLP extract needs to be done, the entire sample must be filtered to collect all the extraction fluid. If the 2 phases cannot be combined, both must be analyzed separately.

5.3.13 After the TCLP extract has been collected, the extract should be aliquoted for analysis and preserved (refrigeration for organics, 1:1 nitric acid to pH <2.0 for metals). Before preservation, it is recommended the pH be recorded.

5.4 TCLP Extraction Procedure for Volatile Organics (VOA):

The Zero-headspace Device (ZHE) is used to obtain TCLP extracts for volatile organics analysis only. It also may be used to separate the solid phase from the liquid phase for VOA's only. The ZHE is made of metal and should not be used for TCLP extractions which will be analyzed for metals or pesticides.

The ZHE device has an approximate internal capacity of 500ml. This means a sample maximum of only 25g of solid may be used due to the need to add extraction fluid #1, equal to 20 times the weight of the solid.
The ZHE is charged with sample only once and the device will not be opened until the final extract has been collected. Repeated filling of the ZHE to obtain 25g of solid is not permitted. The initial filtrate should be weighed and the weight recorded, and then stored until analyzed or recombined with the final extract from the solid.

Although the following procedure allows for particle-size reduction during the conduct of the procedure, this could result in the loss of volatile compounds. If possible, any necessary particle-size reduction should be conducted on the sample as it is being taken. Particle-size reduction should be conducted during the procedure only if there is no other choice.

In carrying out the following steps, do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

5.4.1 Pre-weigh the (evacuated) container which will receive the filtrate (See Section 4.6), and set aside. If using a Tedlar bag, all liquid must be expressed from the device, whether it be for the initial or final liquid/solid separation, and an aliquot taken from the liquid in the bag, for analysis. The containers listed in Section 4.6 are recommended for use under the following conditions.

If waste contains an aqueous liquid phase or does not contain a significant amount of non-aqueous liquid (i.e., <1% of total waste), the Tedlar bag should be used to collect and combine the initial liquid and solid extract. The syringe is not recommended as its use causes unnecessary sample manipulations.

If a waste contains a significant amount of non-aqueous initial liquid phase (i.e., >1% of total waste), the syringe or the Tedlar bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.
If the waste contains no initial liquid phase or has percent dry solids <0.5%, either the Tedlar bag or the syringe may be used.

5.4.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample. Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange)

5.4.3 If the waste is 100% solid (see section 5.2.1), weigh out a representative subsample of the waste, record weight, and proceed to Step 5.4.5.

5.4.4 If the waste was shown to contain <0.5% dry solids (see section 5.2.1), the waste, after filtration is defined as the TCLP extract. Enough of the sample should be filtered so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing >0.5% dry solids, use the percent solids information obtained in Section 5.2.1 to determine the optimum sample size to charge into the ZHE. The appropriate sample size to use is:

For wastes containing <0.5% dry solids, weigh out a representative 500 gram sample of waste and record the weight.

For wastes containing >0.5% dry solids, the amount of waste to charge into the ZHE is determined as follows:

$$\text{weight of waste} = \frac{25}{\%\ \text{solids}} \times 100$$

(Step 7.1 or 7.2)

Weigh out a representative subsample of the waste of the appropriate size and record the weight.

5.4.5 Evaluate the waste to determine if particle-size reduction of the solid portion of the waste is needed prior to extraction. If the solid material within the waste has a surface area per gram of material equal to or greater than 3.1 cm^2, or is smaller than 1 cm in its narrowest dimension (e.g., is capable of passing through a 9.5mm (0.375 inch) standard sieve), particle-size reduction is not required. Proceed to 5.4.7.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost and due to possible contamination of the sample. The use of a ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not required; nor is it recommended.

5.4.6 If the surface area is smaller or the particle-size larger than described in Step 5.4.5, the waste is prepared for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle-size as described in Step 5.4.5.

NOTE: Wastes and appropriate equipment should be refrigerated, if possible, to 4°C prior to particle-size reduction. The means used to effect particle-size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

When the surface area or particle-size has been appropriately altered, proceed to Step 5.4.7.

5.4.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Wastes that settle slowly shall not be centrifuged prior to filtration.

5.4.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions (Appendix #1). Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange out the bottom). Do not attach the extraction collection device to the top plate.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Step 5.4.4, to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10psi (or more if necessary) to force all headspace (into a hood) slowly out of the ZHE device. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If the waste is 100% solid, slowly apply pressure to a maximum of 50psi to force most of the headspace out of the device and proceed to Step 5.4.12.

5.4.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10psi to force the liquid phase into the filtrate collection container. If no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50psi. After each incremental increase of 10psi, if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When liquid low has ceased such that continued pressure filtration at 50psi does not result in any additional filtrate within any 2-minute period, filtration is stopped. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the filtrate collection container.
NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

5.4.10 The material in the ZHE is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. But even after applying pressure filtration, this material will not filter. If this is the

case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% dry solids (see section 5.2.1), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to Step 5.4.15.

5.4.11 The liquid phase may be either analyzed immediately (see Steps 5.4.13-5.4.15) or stored at 4°C under minimal headspace conditions until time of analysis. The weight of extraction fluid #1 to add to the ZHE is determined as follows:

$$\text{weight of extraction fluid} = \frac{20 \times \%\ \text{solids} \times \text{weight of waste filtered}}{100}$$

5.4.12 The following steps detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases.

With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be pre-flushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Pick up the ZHE and physically rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Put 5-10psi behind the piston (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10psi and check all ZHE fittings to ensure that they are closed.
Place the ZHE in the rotary extractor apparatus (if it is not already there) and rotate the ZHE at 30± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction is to occur) shall be maintained at 23 ± 2°C during agitation. Temperature is recorded in the log book. If the temperature is outside 23 ± 2°C, the samples must be re-extracted.

5.4.13 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e.,

no gas release observed), the device is leaking. Replace ZHE O-rings or other fittings, as necessary, check the ZHE for leaking as specified in Appendix 1, and redo the extraction with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR bag) holding the initial liquid phase of the waste, unless doing so would create multiple phases, or unless there is not enough volume left within the filtrate collection container. A separate filtrate collection container must be used in these cases. Filter through the glass fiber filter, using the ZHE device as discussed in Step 5.4.9. All extract shall be filtered and collected if the Tedlar bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase.

NOTE: If the glass fiber filter is not intact following agitation, an in-line glass fiber filter may be used to filter the material within the ZHE.

5.4.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from Step 5.4.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Step 5.4.13 and the initial liquid phase are collectively defined as the TCLP extract.

5.4.15 Following collection of the TCLP extract, the extract should be immediately aliquoted for analysis and stored with minimal headspace at 4°C until analyzed. The TCLP extract will be prepared and analyzed according to the appropriate analytical methods. If the individual phases are to analyzed separately (i.e., are not miscible), determine the volume of the individual phases, conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).

C_1 = The concentration of the contaminant of concern in the first phase (mg/L).

V_2 = The volume of the second phase (L).

C_2 = The concentration of the contaminant of concern in the second phase (mg/L).

5.4.16 The contaminant concentrations in the TCLP extract are compared with the thresholds identified in the appropriate regulations.

6.0 QUALITY CONTROL

6.1 All data, including quality assurance data, should be maintained and available for reference or inspection.

6.2 A minimum of one blank (extraction fluid #1) for every 20 extractions that have been conducted in an extraction vessel shall be employed as a check to determine if any memory effects from the extraction equipment are occurring. One blank should also be employed for every new batch of leaching fluid that is made up.

6.3 For each analytical batch (up to twenty samples), a matrix spike must be performed. Addition of matrix spikes should occur once the TCLP extract has been generated and preserved. If during the preservation, a precipitate is noticed, then the spike should be added before the extract is preserved.The purpose of the matrix spike is to monitor the adequacy of the analytical methods used on the TCLP extract and for determining if matrix interferences exist in analyte detection.

6.4 All quality control measures described in the appropriate analytical methods shall be followed.

6.5 The method of standard addition shall be employed for each metallic analyte if: (1) recovery of the compound from the TCLP extract spike is less than 50%, or (2) if the concentration of the constituent measured in the extract is between 80-100% of the appropriate regulatory threshold. If the matrix spike fails the above two categories and more than one extract is being run, all samples of the same waste (up to twenty samples), the method of standard addition should be performed on each sample.

6.6 Samples must undergo TCLP extraction within the following time period after sample receipt: Volatiles, 14 days; Semi-Volatiles, 14 days; Mercury, 28 days; and other Metals, 180 days. Extraction of the solid portion of the waste should be initiated as soon as possible following initial solid/liquid separation. TCLP extracts shall be analyzed after generation and preservation within the following periods: Volatiles, 14 days; Semi-Volatiles (after preparative extraction), 40 days; Mercury, 28 days; and other Metals, 180 days.

7.0 SAFETY

7.1 Technicians performing this procedure should wear a lab coat, gloves and safety glasses.

7.2 Positive pressure filtration should take place in a sink or hood in the event a filter bursts and sprays sample out of the bottom opening.

8.0 WASTE MANAGEMENT

8.1 Extracts should be stored in the digestion lab for 6 months. Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

9.0 REPORTING REQUIREMENTS

9.1 Once the extraction is finished and the extract is filtered, return to sample receiving for a job code assignment.

Fill out Metals Digestion Lab TCLP log form enclosed as Attachment 2 for all TCLP Extractions.

10.0 DEFINITIONS

10.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

FIGURE 1.A
TCLP FLOWCHART

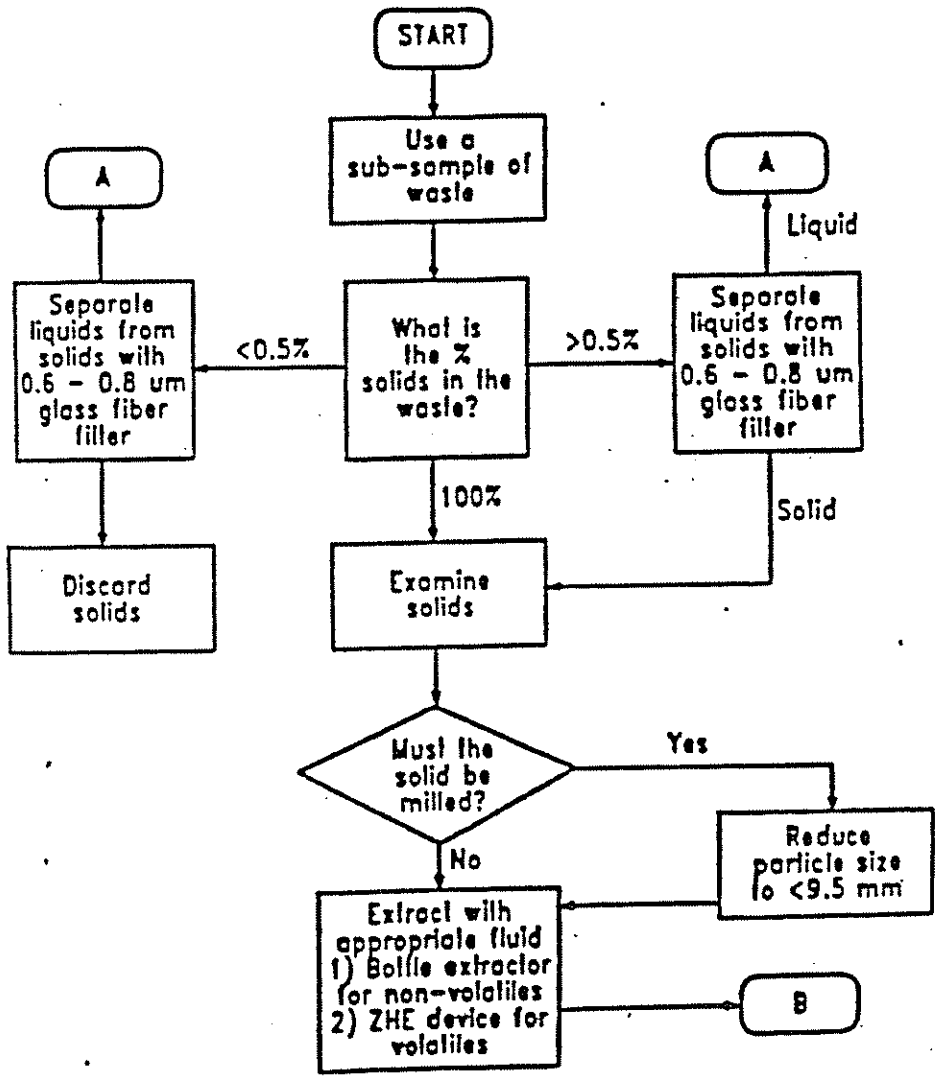
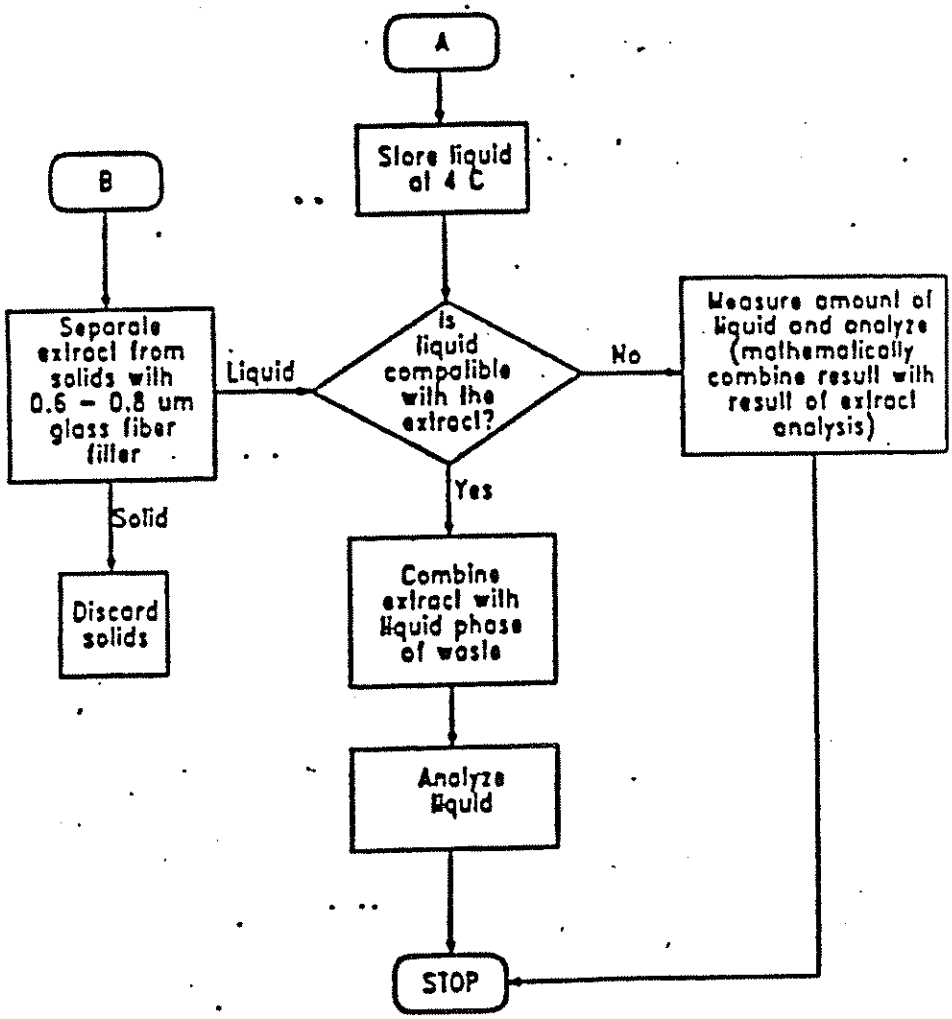


FIGURE 1.B
TCLP FLOWCHART

METHOD 1311 (CONTINUED)

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE

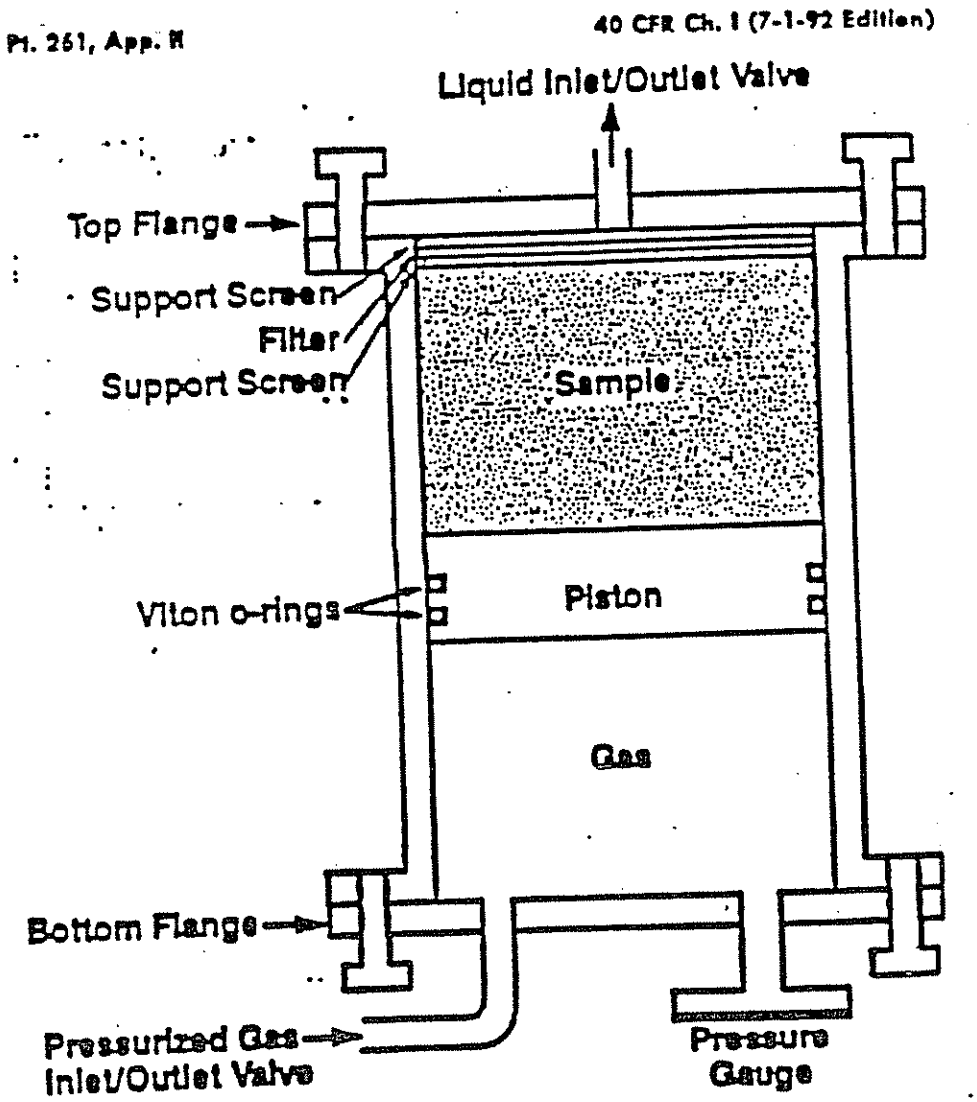


[55 FR 26987, June 29, 1990]

TABLE 1
VOLATILE CONTAMINANTS

Compound	CAS NO.
Acetone	67-64-1
Acrylonitrile	107-13-1
Benzene	71-43-2
n-Butyl alcohol	71-36-6
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,1,2-trifluorcethane	76-13-1
Vinyl chloride	75-01-4
Xylene	1330-20-7

FIGURE 3.
ZERO-HEADSPACE EXTRACTION VESSEL



Appendix #1

ASSOCIATED DESIGN & MANUFACTURING CO.
814 NORTH HENRY STREET
ALEXANDRIA, VA 22314
PHONE: (703) 549-5999

LOADING PROCEDURE FOR ZERO HEAD SPACE EXTRACTOR VESSEL

1) Place pressure vessel (Item 4) air gauge side down in the chair (Item 10).

2) Remove the end flange with the test valve (Item 8).

3) Remove the filter pack holder screen (Item 7).

4) Open the air bleed valve (Item 12), wet piston (Item 5) and o-ring (Item 14) then push the piston into the cylinder, allowing enough room for sample.

5) Place the sample to be extracted into the vessel.

6) Install the filter pack and screen (Item 7) over o-ring, install end cap (Item 8) and install four (4) knobs. Tighten opposing screws evenly until an even gap between end cap and body is achieved.

7) Close the test fitting valve (Item 9).

8) Close the air bleed valve (Item 12).

9) Attack a source of compressed air or nitrogen to the quick disconnect fitting (50psi max.) (Item 2) and pressurize vessel to 1-10psi. (Note: if compressed air is used, a line filter should be installed to prevent dirt or rust from scoring the walls of the Zero Head Space Extractor Vessel.)

10) Check for leaks by observing pressure gauge drop.

11) Crack test valve (Item 9) as required to allow entrapped air to escape. Close the valve as soon as traces of the liquid are seen. If necessary increase pressure in 10psi increments until appearance of liquid is noted.

12) Insert teflon leur lock fitting (Item 11) to test valve and lock. Attach syringe to the test valve (Item 9).

13) Open the test valve (Item 9) until all of the liquids have been removed from the test sample. Increase air pressure in 10psi increments to 50psi.

14) Close the test valve (Item 9) and remove collection container from the vessel.

15) Remove teflon leur lock fitting (Item 11) and insert tube from a source of fresh extraction fluid to the test valve (Item 9). Using a suitable means of introducing the fluid by a positive displacement pump or cylinder, pump the required quantity of fluid into the vessel and close the test valve (Item 9). (Suggested source for pump is: Fluid Metering, Inc. (516) 922-605 Model #RP-SY-2CKC).

16) Pressurize vessel (piston side) to 5-10psi.

17) Place the pressure vessel in the rotary extractor and rotate per required time.

18) Following rotation of the vessel for the prescribed time and at the prescribed temperature, perform a second filtration as above. The analysis may now be performed. Repeat steps 12 through 14 and analyze the collected liquid.

CAUTION: NEVER ATTEMPT TO DIS-ASSEMBLE THE VESSEL UNTIL ALL AIR PRESSURE HAS BEEN RELEASED BY MEANS OF THE AIR BLEED VALVE.

CLEANING PROCEDURE FOR ZERO HEADSPACE EXTRACTOR VESSEL

The internal piston and cylinder are precisely machined, combined with the o-rings on the piston, providing a tight seal. This seal is required to eliminate any seepage of samples. Thorough cleaning is required after each use to maintain a proper seal.

1) Remove the end caps and rinse out the cylinder (Item 4) and the piston (Item 5).

2 Using the plastic rod supplied, insert the rod in the cylinder (Item 4) locating the rod on the center of the piston (Item 5) and push the piston out.

NOTE:IF THE PISTON WILL NOT MOVE, TAP THE END OF THE ROD LIGHTLY WITH A RUBBER MALLET.

WARNING! DO NOT USE AIR TO MOVE PISTON!!

3) Remove the o-ring (Item 14) from the piston (Item 5) by rolling the o-ring out of their grooves. If the o-rings are stuck in the grooves wet them down with water and pinch the o-rings between two fingers to loosen them up and then roll the off.

4) Wash cylinder (Item 4), piston (item 5), filter plate (Item 8) and o-rings (Item 14) in a soapy-water solution and rinse with distilled water.

NOTE:IF VESSEL IS HEAVILY CONTAMINATED, PASSIVATE PER MIL-STD-QQ-P-35B, TYPE II, BY A LOCAL PLATING SHOP. THE FOLLOWING PARTS SHOULD BE PASSIVATED:
PISTON (ITEM 5)
CYLINDER (ITEM 4)
FILTER PLATE (ITEM 8)
AFTER PASSIVATION, RINSE IN DISTILLED WATER.

CAUTION: DO NOT PASSIVATE AIR SIDE PLATE WITH GAUGES AND VALVES.

5) Wet the o-rings (Item 14) with distilled water and roll them back on the piston (Item 5), wet the cylinder (Item 4) with distilled water and install the piston being careful not to pinch the o-rings.

6) Re-assemble per instructions on loading pressure vessel.

ASSOCIATED DESIGN & MANUFACTURING CO.
814 NORTH HENRY STREET
ALEXANDRIA, VA 22314
PHONE: (703) 549-5999

ITEM NO.	PART NUMBER	DESCRIPTION
1	5041-0001	PRESSURE GAUGE
2	5040-0003	QUICK CONN. (MALE)
3	6001-0002	TOP PLATE AIRSIDE
4	6001-0004	BODY
5	6001-0008	PISTON
6	6001-0007	COLLAR CLAMP
7	5044-0001	FILTER PACK WITH .7 MICRON FILTER
8	6001-0003	FILTER PLATE
9	5040-0001	TEST VALVE
10	6001-0009	CHAIR (SET UP)
11	6001-0014	LUER LOCK
12	5040-0004	VALVE, AIR PRESSURE
13	5030-0003	O-RING, BODY (QTY:3)
14	5030-0002	O-RING, PISTON (QTY:2)
15	5030-0001	O-RING, FILTER (QTY:1)
16	5040-0002	QUICK CONN. (FEMALE)
17	6001-0011	SCREEN

Attachment 1

The following constituents are now regulated under the Toxicity Characteristic rule. Waste generators must determine the levels present in their waste sample extract or leachate, based either on their knowledge of their processes or by application of the TCLP.

New Constituents/Regulatory levels

Benzene . . . 0.50mg/l
Carbon tetrachloride . . . 0.50mg/l
Chlordane . . . 0.03mg/l
Chlorobenzene . . . 100.0mg/l
Chloroform . . . 6.0mg/l
m-Cresol . . . 200.0mg/l
o-Cresol . . . 200.0mg/l
p-Cresol . . . 200.0mg/l
1,4-Dichlorobenzene . . . 7.5mg/l
1,2-Dichloroethane . . . 0.50mg/l
1,1-Dichloroethylene . . . 0.70mg/l
2,4-Dinirotoluene. . . 0.13mg/l
Heptachlor (and its hydroxide) . . . 0.008mg/l
Hexachloro-1,3-butadiene . . . 0.5mg/l
Hexachlorobenzene . . . 0.13mg/l
Hexachloroethane . . . 3.0mg/l
Methyl ethyl ketone . . . 200.0mg/l
Nitrobenzene . . . 2.0mg/l
Pentachlorophenal . . . 100.0mg/l
Pyridine . . . 5.0mg/l
Tetrachloroethylene . . . 0.5mg/l
Trichloroethylene . . . 0.5mg/l
2,4,5-Trichlorophenol . . . 400.0mg/l
2,4,6-Trichlorophenol . . . 2.0mg/l
Vinyl chloride . . . 0.20mg/l

Old EP Constituents/Regulatory levels

Arsenic . . . 5.0mg/l
Barium . . . 100.0mg/l
Cadmium . . . 1.0mg/l
Chromium . . . 5.0mg/l
Lead . . . 5.0mg/l
Mercury . . . 0.2mg/l
Selenium . . . 1.0mg/l
Silver . . . 5.0mg/l
Endrin . . . 0.02mg/l
Lindane . . . 0.4mg/l
Methoxychlor . . . 10.0mg/l
Toxaphene . . . 0.5mg/l
2,4-Dichlorophenoxycetic acid . . . 10.0mg/l
2,4,5-Trichlorophenoxypropionic acid . . . 1.0mg/l

Appendix B
Field SOPs

BROWN & ROOT ENVIRONMENTAL	STANDARD OPERATING PROCEDURES	Number CT-04	Page 1 of 6
		Effective Date 03/01/96	Revision 0
		Applicability B&R Environmental, NE	
		Prepared Risk Assessment Department	
Subject SAMPLE NOMENCLATURE		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this document is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix.
- Sorting of data by depth.
- Maintenance of consistency (field, laboratory, and data base sample numbers).
- Accommodation of all project-specific requirements on a global basis.
- Accommodation of laboratory sample number length constraints (10 characters).

2.0 SCOPE

The methods described in this procedure shall be used consistently for all projects requiring electronic data handling managed by personnel located in the Northeast Region of Brown & Root Environmental (Pittsburgh, Wayne, Holt, and Wilmington) and for any large contracts managed by the Northeast Region (e.g., NORTHDIV CLEAN, SOUTHDIV CLEAN, ARCS I, ARCS III, etc.). Smaller projects (as determined by Project Manager) are outside the scope of this SOP.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Program Manager - It shall be the responsibility of the Program Manager (or designee) to inform contract-specific Project Managers of the existence and requirements of this Standard Operating Procedure.

Project Manager - It shall be the responsibility of the Project Manager to determine the applicability of this Standard Operating Procedure based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the sample nomenclature is thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and is consistent with this Standard Operating Procedure if relevant. It shall be the responsibility of the project manager to ensure that the Field Operations Leader is familiar with the sample nomenclature system.

Field Operations Leader - It shall be the responsibility of the Field Operations Leader to ensure that all field technicians or sampling personnel are thoroughly familiar with this Standard Operating Procedure and the project-specific sample nomenclature system. It shall be the responsibility of the Field Operations Leader to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

5.0 PROCEDURES

5.1 Introduction

The sample numbering system consists of 12 distinct alpha-numeric characters, only 10 of which will be provided to the laboratory on the sample labels and chain-of-custody forms. The sample number provided to the lab shall be as follows where "A" indicates "alpha," "N" indicates "numeric," and "E" indicates "either"):

$$\overline{E}\ \overline{E}\ \overline{E}\ \overline{A}\ \overline{A}\ \overline{E}\ \overline{E}\ \overline{E}\ \overline{N}\ \overline{N}$$

Once the analytical results are received from the laboratory the sample number will be revised by a subroutine such that the sample number is more user friendly (i.e., dashes will be inserted). The sample number will then appear as follows:

$$\overline{E}\ \overline{E}\ \overline{E}\ -\ \overline{A}\ \overline{A}\ -\ \overline{E}\ \overline{E}\ \overline{E}\ -\ \overline{N}\ \overline{N}$$

If multiple sampling events occur (or are planned) for a given matrix, a subroutine within the database will be used to append two additional characters such that the sample number will appear as follows:

$$\overline{E}\ \overline{E}\ \overline{E}\ -\ \overline{A}\ \overline{A}\ -\ \overline{E}\ \overline{E}\ \overline{E}\ -\ \overline{N}\ \overline{N}\ -\ \overline{N}\ \overline{N}$$

Site Type Location Depth Round

5.2 Sample Number Field Requirements

The various fields in the sample number will include the following:

- Site Identifier
- Sample Type
- Sample Location
- Sample Depth Indicator
- Sampling Round

The site identifier must be a three-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary since many facilities/sites have multiple individual sites, SWMUs, operable units, etc.

The sample type must be a two-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be a three-character field (alpha, numeric, or a mixture).

The depth field must be provided for all samples, regardless if it is strictly applicable (as discussed in Section 5.3).

The sampling round is optional, but, if provided, must be two numeric characters.

5.3 Example Sample Field Designations

Examples of each of the fields are as follows:

Site Number - Examples of site numbers/designations are as follows:

A01 - Area of Concern Number 1
125 - Solid Waste Management Unit Number 125
000 - Base or Facility Wide Sample (e.g., upgradient well)
BBG - Base Background

The examples cited are only suggestions. Each Project Manager (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample Type - Examples of sample types are as follows:

AS - Air Sample
BS - Biota Sample (See Note)
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
ID - Investigation Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample
SG - Soil Gas Sample
SP - Seep Sample
SS - Surface Soil Sample
SU - Subsurface Soil Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WI - Wipe Sample
WP - Well Point Sample
WS - Waste/Sludge Sample

Note: The biota sample designation may be contingent upon the type of biota sampled (e.g., BL - Lobster; BF - Finfish; BC - Clam; BO - Oyster). Numerous other examples can be cited but will be site-specific.

This field will also be used to designate field Quality Control Samples, as follows:

TB - Trip Blank
FB - Field Blank
RB - Rinsate Blank (Equipment Blank)
BB - Bottle Blank
AB - Ambient Condition Blank

Field quality control samples should be numbered sequentially (e.g., RB-001; FB-010, etc.).

Filtered/unfiltered surface water or groundwater samples shall be handled in an separate manner, as subsequently discussed.

Location - Examples of the location field are as follows:

A01 - Grid node A1
001 - Monitoring Well 1

It is important that consistency be maintained with respect to the use of the characters "0" and O. Data base subroutines will not sort correctly if a mixture are used (e.g, AO1 and A02).

Depth - Formerly, depth specifications were indicated with a four digit field (e.g., 0002 - 0 to 2 feet). While this is effective for depth sorting, it is difficult to include this level of detail in a 10-character lab number (FormMaster limitations). In addition, this approach will not accommodate non-integer depths (e.g., 2.5 feet to 4.5 feet).

Based on such potential problems, the following approach shall be used: Sample depths will simply represent the horizon from which the sample was obtained: For example, if ten split-spoon samples are collected from a boring, they will be numbered 01 through 10. The sample log sheet will be used to record the specific depth of the sample, and this information will be entered in a separator field in the data base.

Similar nomenclature will be used for depth-specific surface water and sediment samples, etc. If no depth information is required (e.g., groundwater samples), the field must still be filled (e.g., Ø, Ø).

This field will also be used for the designation of filtered and unfiltered samples. An unfiltered groundwater sample shall be designated as U0, if and only if, a corresponding filtered sample is collected. Such as sample shall be designated as F0.

Sampling Round - The sampling round field is straightforward. It can range from 01 to 99.

5.4 Example Sample Numbers

Examples of complete sample numbers (field/data base versus laboratory) are as follows:

Field/Data Base ID	Lab ID	Description
101-SB-A01-01	101SBA0101	The first sample (e.g., 0 to 2 feet) from soil boring A01 (grid) at Site 101.
101-SB-A01-02	101SBA0102	The second sample from boring A01 (could be the next depth interval or a duplicate of 101-SB-A01-01).
125-MW-001-01-01	125MW00101	A groundwater sample from monitoring well MW001 (first sampling round)
125-MW-001-02-01	125MW00102	A duplicate groundwater sample from monitoring well MW001 (first sampling round)
130-MW-003-U1-01	130MW003U1	An unfiltered groundwater sample from monitoring well MW003 (first sampling round)
130-MW-003-F1-01	130MW003F1	A filtered groundwater sample from monitoring well MW003 (first sampling round)
137-RB-001-00-01	137RB00100	The first rinsate blank collected at site 137.
137-TB-004-00-02	137TB00400	The fourth trip blank collected during the second sampling event at Site 137.
155-SW-003-01-01	155SW00301	A surface water sample collected from the surface of a pond at Site 155.
155-SW-003-02-01	155SW00302	A surface water sample collected from the bottom of the water column in a pond at Site 155.

Corrective Action Form

Person initiating corrective action _____ Date _____

Description of problem and when identified: _____

_____

_____

Cause of problem, if known or suspected:

_____

_____

_____

_____

Sequence of Corrective Action (CA): (including date implemented, action planned and personnel/data affected)

_____

_____

_____

_____

_____

_____

CA implemented by: _____ Date: _____

CA initially approved by: _____ Date: _____

Follow-up date: _____

Final CA approved by: _____ Date: _____

Information copies to:

Anita Dodson, CH2M HILL Program Chemist

Navy QAO

STANDARD OPERATING PROCEDURE

Direct-Push Groundwater Sample Collection

I. Purpose

To provide a general guideline for the collection of groundwater samples using direct-push (e.g., Geoprobe®) sampling methods.

II. Scope

Standard direct-push (e.g., Geoprobe®) groundwater sampling methods.

III. Equipment and Materials

- Truck-mounted hydraulic percussion hammer.
- Direct-push (e.g., Geoprobe®) sampling rods and slotted lead rod
- Polyethylene sampling tubing and stainless steel foot valve
- Pre-cleaned sample containers
- Clean latex or surgical gloves.

IV. Procedures and Guidelines

1. Decontaminate slotted lead rod and other downhole equipment in accordance with SOP *Decontamination of Personnel and Equipment*.
2. Drive slotted steel lead rod to the desired sampling depth using the truck-mounted hydraulic percussion hammer.
3. Insert the stainless steel foot valve into the end of the polyethylene sampling tubing and insert tubing through the rods.
4. Fill all sample containers, beginning with the containers for VOC analysis.
5. Remove polyethylene sampling tubing from the rods. Remove the foot valve and discard polyethylene tubing.
6. Backfill borehole at each sampling location with grout or bentonite and repair the surface with like material (bentonite, asphalt patch, concrete, etc.), as required.

V. Key Checks and Items

- Verify that the hydraulic percussion hammer is clean and in proper working order.
- Ensure that the direct-push operator thoroughly completes the decontamination process between sampling locations.
- Ensure that the slotted lead rod has been inserted to the desired sampling depth.
- Verify that the borehole made during sampling activities has been properly backfilled.

STANDARD OPERATING PROCEDURE

Direct-Push Soil Sample Collection

I. Purpose

To provide a general guideline for the collection of soil samples using direct-push (e.g., Geoprobe®) sampling methods.

II. Scope

Standard direct-push (e.g., Geoprobe®) soil sampling methods.

III. Equipment and Materials

- Truck-mounted hydraulic percussion hammer.
- Sampling rods
- Sampling tubes and acetate liners (if desired)
- Pre-cleaned sample containers and stainless-steel sampling implements
- Clean latex or surgical gloves.

IV. Procedures and Guidelines

1. Decontaminate sampling tubes and other non-dedicated downhole equipment in accordance with SOP *Decontamination of Personnel and Equipment*.

2. Drive sampling tube to the desired sampling depth using the truck-mounted hydraulic percussion hammer. If soil above the desired depth is not to be sampled, first drive the lead rod, without a sampling tube, to the top of the desired depth.

3. Remove the rods and sampling tube from the borehole and remove the sample from the tube.

4. Fill all sample containers, beginning with the containers for VOC analysis, using a decontaminated or dedicated sampling implement.

5. Decontaminate all non-dedicated downhole equipment (rods, sampling tubes, etc.) in accordance with SOP *Decontamination of Personnel and Equipment*.

6. Backfill borehole at each sampling location with grout or bentonite and repair the surface with like material (bentonite, asphalt patch, concrete, etc.), as required.

V. Key Checks and Items

1. Verify that the hydraulic percussion hammer is clean and in proper working order.

2. Ensure that the direct-push operator thoroughly completes the decontamination process between sampling locations.

3. Verify that the borehole made during sampling activities has been properly backfilled.

EQUIPMENT CLEANING

Bailers, sampling pumps, purge pumps, and other non-dedicated purging or sampling apparatus will be cleaned before and after sampling each well. Factory new and sealed disposable bailer may be used for sampling, but may not be reused. Thermometers, pH electrodes, and SEC probes that will be used repeatedly will be cleaned before and after sampling each well and at any time during sampling if the object comes in contact with foreign matter.

Purged waters and solutions resulting from cleaning of purging or sampling equipment will be collected and stored properly for future disposal by the client, unless other arrangements have been made.

Cleaning of reusable equipment that is not dedicated to a particular well will consist of the following:

Bailers - the inside and outside of bailers will be cleaned in a solution of laboratory-grade detergent and potable water, followed by a rinse with deionized (DI) water. They may also be steam-cleaned, followed by a DI water rinse. If samples are to be collected for metals analysis, the Teflon® bailer may be rinsed with a pH2 nitric acid solution followed by a double DI rinse.

Purge Pumps - All downhole, reusable portions of purge pumps will be steam-cleaned on the outside. If the pump does not have a backflow check valve, the inside of the pump and tubing also should be steam-cleaned by pumping a laboratory-grade detergent and potable water solution through the system followed by a potable water rinse, or by steam-cleaning.

Water Quality Meters - All meters will be cleaned by rinsing the probe portions in DI water, and allowing to air dry.

Bailer Tripod - The tripod cable will be steam cleaned or rinsed with DI water.

Sample bottles and bottle caps will be cleaned by the subcontracted laboratory using standard EPA-approved protocols. Sample bottles and bottle caps will be protected from contact with solvents, dust, or other contamination. Sample bottles will not be reused.

REFERENCES

Barcelona, M.J., et al., 1994, Reproducible Well-Purging Procedures and VOC Stabilization Criteria for Ground-Water Sampling: Groundwater, January-February.

Kearl, P.M., et al., 1994, Field Comparison of Micropurging vs. Traditional Ground Water Sampling: Ground Water Monitoring Review, Fall.

FIELD MEASUREMENTS

Field measurements of temperature, pH, and SEC will be performed on aliquots of groundwater that will not be submitted for laboratory analysis. Field water quality measurements and instrument calibration details will be recorded on the WELL SAMPLING AND/OR DEVELOPMENT RECORD.

TEMPERATURE MEASUREMENTS

Temperature measurements will be made with a mercury-filled thermometer or an electronic thermometer, and all measurements will be recorded in degrees Celsius.

pH MEASUREMENT

The pH measurement will be made as soon as possible after collection of the sample, generally within a few minutes. The pH will be measured by immersing the pH probe into an aliquot of groundwater.

The pH meter will be calibrated at the beginning of and once during each sampling day and whenever appropriate, in accordance with the equipment manufacturer's specifications, as outlined in the instruction manual for the specific pH meter used. Two buffers (either pH-4 and pH-7, or pH-7 and pH-10, whichever most closely bracket the anticipated range of groundwater conditions) will be used for instrument calibration.

SPECIFIC ELECTRICAL CONDUCTANCE MEASUREMENT

Specific electrical conductance (SEC) will be measured by immersing the conductivity probe into an aliquot of groundwater. The probes used should automatically compensate for the temperature of the sample. Measurements will be reported in units of micro-Siemens (μS) per square centimeter (equivalent to micromhos or μmhos) at 25 degrees Celsius.

The SEC meter will be calibrated at the beginning and once during each sampling day in accordance with the equipment manufacturer's specifications, as outlined in the instruction manual for the SEC meter used. The SEC meter will be calibrated with the available standardized potassium chloride (KCI) solution that is closest to the SEC expected in groundwater below the site.

GPL LABORATORIES
7210A Corporate Ct.
Frederick, MD 21703
301-694-5310

CHAIN OF CUSTODY

GPL Project Manager:	Client Name:	Phone:	
Project Name:	Address:	Cell:	
Purchase Order:	City, State	Zip Code	

Comments: H=Hold Analysis Request X=Analyze

Preservatives and Containers

Sample Information | **Methods for Analysis** | **RUSH**

No.	Client Name, address and phone #	Date Sampled	Time Sampled	Matrix	Sampler's Initials																QC (MS/MSD)	TOTAL BOTTLES
1							X	X	X	X	X	X	X	X	X	X		X					4
2							X									.							
3																							
4																							
5																							
6																							
7																							
8																							
9																							
10																							
11																							
12																							

Sample Matrix: WG= Groundwater; SO= Soil; WS= Surface Water; AA= Ambient Air; WQ= Water Quality

Total number of samples 4

Relinquished By:	**Date:**	**Time:**
Received By:	**Date:**	**Time:**
Relinquished By:	**Date:**	**Time:**
Received By:	**Date:**	**Time:**
Relinquished By:	**Date:**	**Time:**

For Lab Use

GPL WORK ORDER #:_____

COOLER RECEIPT CONDITION

CLICK HERE TO GET TO ATTACHMENTS

Waste Management: Analysis and Characterization Enterprise Standard Operating Procedure HSE-408

1.0 Purpose

This Enterprise Standard Operating Procedure (SOP) describes procedures for analyzing and characterizing waste streams generated on CH2M HILL projects. It is designed to be used in conjunction with HSE-413, Waste Management Planning.

2.0 Scope and Application

2.1 Scope

Managing waste from construction, demolition, remediation, or other field activities at CH2M HILL project sites is an important aspect of our business. This Waste Analysis and Characterization SOP lists the proper procedures to determine the regulatory status of wastes and is applicable to project activities that generate waste.

2.2 Application

This SOP applies Enterprise Wide to all CH2M HILL Legal Entities and Business Groups, their employees, subcontractors and their lower-tier subcontractors that operate in the United States (US) and internationally.

Some state environmental or OSHA programs may have more stringent requirements. State regulations may be found under Regtools. Contact the appropriate Responsible Environmental Manager (REM) to address these specific requirements. This SOP should be used as a starting point for international operations, but country-specific health, safety and environmental (HSE) regulations shall prevail, and a country-specific SOP should be developed to comply with these specific HSE regulations.

2.3 Applicable Enterprise SOPs

Applicable Enterprise Standards of Practice and Standard Operating Procedures that are applicable to this SOP are as follows:

- HSE-413, Waste Management: Planning

3.0 Definitions

3.1 Chain-of-Custody Form

A form that documents the transfer of custody and responsibility from the sampling personnel to the laboratory. This form also documents the identification of the samples and the analyses requested.

3.2 Characterization

Characterization is the process where waste constituent concentrations are identified and compared to legal requirements or threshold levels that affect waste disposal methods. The generator of the waste is required by law to perform this process. The process may include evaluation of historical or MSDS information and/or analytical sampling and analysis.

3.3 Custody Seal

A label with adhesive backing that is placed on the sample and/or shipping container to enable detection of sample tampering.

3.4 Detection Limit

A detection limit is the expression for the minimum value reported by the laboratory for an analytical test method for a given constituent. Other terms that may be used in consultation with the laboratory include practical quantitation limit (PQL), sample quantitation limit (SQL), method detection limit (MDL), and project-specific reporting limit (PRL).

3.5 Generator

A generator is any person whose act or process produces hazardous waste or whose act first causes hazardous waste to become subject to regulation.

3.6 Logbook

A bound field notebook, with consecutively pre-numbered pages, that is clearly identified with the name of the project activity, the person responsible for maintenance of the notebook, and the beginning and ending dates of the entries.

3.7 Regulatory Level

A regulatory level is the legal standard or threshold level that determines a waste characterization.

3.8 Sample Label

A label with adhesive backing that is affixed to each individual container containing the identification of the sample, the preservatives applied to the sample, analytical method to be used, the sampling date and time, and the name of the sampler.

3.9 Transporter

A transporter is engaged in the off-site transportation of waste by air, rail, highway, or water.

4.0 Roles and Responsibilities

4.1 Project Delivery Manager (PDM)

The Project Delivery Manager (PDM) is responsible for ensuring that Project Managers are aware of the policies and procedures in this SOP.

4.2 Project Manager (PM)

The Project Manager (PM) is responsible for ensuring the project is implemented in compliance with environmental requirements. The PM should work with the REM to identify CH2M HILL responsibilities, costs, and plan implementation.

4.3 Responsible Environmental Manager (REM)

The REM assists the project manager in ensuring that the project complies with environmental laws and regulations by identifying issues and resources to meet project needs.

4.4 Safety Coordinator (SC)

The Safety Coordinator is familiar with project plans, including wastewater and storm water requirements, and implements the plans in the field.

4.5 Field Sampling Personnel

Field sampling personnel are responsible for following these procedures and those listed in the Sampling and Analysis Plan during sampling activities, including recording pertinent data into the logbook to satisfy project requirements.

4.6 Project Quality Control Manager

The Project QC Manager is responsible for ensuring that all field personnel follow these procedures and complete the sampling records described in this SOP. The Project QC Manager is also responsible for the following:

- Reviewing logbook entries to ensure that the sampling event is properly and completely documented, and that each page is dated and signed
- Verifying that the Chain-of-Custody (COC) Forms have been prepared completely and properly
- Verifying that the sample labels correspond to the samples listed on the COC form, and
- Verifying that the samples collected and the analyses requested on the COC match the Sampling and Analysis Plan (SAP) and the Laboratory's Purchase Order (PO) and correct discrepancies

5.0 Procedures

CH2M HILL requires managing wastes in compliance with applicable legal requirements. Waste characterization is the client's legal responsibility. CH2M HILL will not sign documentation (e.g., manifests) that suggests CH2M HILL is assuming the client's waste characterization responsibility.

5.1 General Waste Characterization Information

As discussed in HSE-413, Waste Management Planning, it is the client's responsibility to characterize all waste streams. CH2M HILL may assist a client with waste characterization if specified in the project scope of work and all necessary approvals are obtained (see CH2M HILL Hazardous Waste Policy).

Waste stream characterization should be documented in the project file. The following should be considered when assisting a client with waste characterization:

- Assume waste is hazardous until proven non-hazardous
- Characterize before waste is generated (client may be able to do this using generator knowledge)
- Estimate waste volumes
- Identify disposal facility sampling and analytical requirements

Waste characterization requirements vary depending upon where the project is located. Consult the REM for assistance.

5.2 Waste Sampling and Analysis

Sampling and analysis may be required when a waste stream cannot be properly characterized based on historical or process knowledge, or is required pursuant to a legal requirement. This section outlines the key elements for identifying analytical requirements for managing wastes. If analysis is required, the following elements should be considered:

- Analytical methods and reporting format
- Sampling protocols

5.2.1 Identify Analytical Test Methods

Waste characterization may require multiple analytical test methods, depending upon the potential composition and legal requirements. Analytical test methods will depend upon the following considerations:

- The nature and quantity of waste
- Legal requirements for transporting, treating, and disposing of the waste
- Analytical method detection limits
- Analysis required by the disposal facility

Waste Nature and Quantity

Evaluate the historical and process information related to waste generation. This information is usually obtained through the client or a review of the MSDS. An understanding of the known and expected constituents will help determine what analytical parameters are required. The quantity of waste to be generated will also affect what types of samples may be used (e.g., composite or grab samples).

Legal Requirements

Legal requirements for waste analysis, transport, treatment, and disposal vary depending upon where the project is located. Consult the REM for state and provincial requirements.

Detection Limits

Consult with the laboratory to identify the detection limits of the proposed test methods and review the required levels that must be met. The analytical method should satisfy all the analytical requirements necessary to characterize and safely transport, treat, and dispose of the waste.

Disposal Facility Requirements

Many disposal facilities have waste acceptance criteria that are in addition to the regulatory waste characterization requirements (e.g., Paint Filter Liquids Test [EPA Method 9095]). The waste acceptance criteria may affect the test method or number of samples required per volume of waste. Contact REM to evaluate proposed disposal facilities (including landfill and wastewater treatment plants) and to identify the waste acceptance criteria for each project waste stream.

5.3 Sampling

A sampling plan should be developed to ensure analysis is based on representative samples. Consult the REM and project chemist to determine sampling requirements.

Standards for documenting field sampling activities, labeling the samples, documenting sample custody, and completing the COC form are provided in this procedure. The standards presented in this section shall be followed to ensure that samples collected are maintained for their intended purpose and that the conditions encountered during field activities are documented. This procedure shall apply to all sample collection conducted during CH2M HILL field activities.

5.3.1 Logbook

The field logbook serves as the primary record of field activities. Entries shall be made chronologically and in sufficient detail to allow the writer or a reviewer to reconstruct each day's events. Each day's activities should be signed and dated. Any changes to the Sampling and Analysis Plan shall be documented in the field logbook. If changes need to be made to the sampling procedures or analysis (e.g., not enough recovery in a well to obtain enough groundwater sample for complete analysis), the field staff shall contact the PM (in consultation with the local REM) to determine the appropriate action (e.g., which analyses will be omitted). Field logs such as soil boring logs and groundwater sampling logs should also be used.

5.3.2 Custody Procedures

For samples intended for chemical analysis, sample custody procedures shall be followed through sample collection, transfer, analysis, and final sample disposal to ensure that the integrity of the samples are maintained. Custody of samples shall be maintained in accordance with the sample custody procedures described below.

A sample is considered to be in custody if:

- It is in one's actual physical possession or view
- It is in one's physical possession and has not been tampered with (i.e., it is under lock or official seal)
- It is retained in a secured area with restricted access

- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal

5.3.3 Sample Labels

A sample label shall be affixed on each individual sample container. Clear tape should be placed over each label to prevent the labels from tearing off, falling off, being smeared, and to prevent loss of information on the label. See Attachment 1.

5.3.4 Custody Seals

A minimum of two custody seals shall be placed on each container (bottle/jar/vial) immediately after sample collection or on sample shipping containers (coolers) prior to shipment of the samples. Custody seals will be placed in such a manner that they must be broken to open the sample container (bottle/jar/vial) or sample shipping containers (coolers).

The seals shall be placed over the left and right sides of the container's (cooler's) cover or be placed across the top of each individual sample container (bottle/jar/vial). Each custody seal must be signed and dated. Clear tape should be placed over each custody seal to prevent the custody seals from easily tearing or falling off. See Attachment 2.

5.3.5 Chain-of-Custody Form Instructions

The COC form completion procedures are crucial in properly transferring the custody and responsibility of samples from field personnel to the laboratory. This form also is important for accurately and concisely requesting analyses for each sample; it is essentially a release order from the analysis subcontract. Instruction for properly completing the form and contained in Attachment 3: Chain of Custody Form and Instructions.

Field personnel shall also log individual samples onto COC forms when a sample is collected. These forms may also serve as the request for analyses.

The samplers shall sign the COC form signifying they were the personnel who collected the samples. The COC form shall accompany the samples from the field to the laboratory. If the samples are given to another person for shipment, the sampler must transfer custody of the samples to that person. This transfer shall be indicated on the COC form. When the sample shipping container (cooler) is ready for shipment to the analytical laboratory, the name of the shipping company, along with the tracking number, shall be documented on the COC form, indicating a transfer of custody from the field to the shipping company. One copy of the COC form shall be retained by the field while the original COC form is placed in a plastic zip sleeve and taped to the inside of the sample shipping container (cooler) cover.

Each sample shipping container (cooler) must be associated with a unique COC form.

The electronic version of the COC forms shall be sent by e-mail or fax to both the laboratory and the project chemist in order to alert them that the samples are in transit to the laboratory.

5.3.6 Records

The COC form shall be sent by email or faxed to the project chemist and the laboratory upon shipment of the sample shipping containers (coolers). The original COC form shall be submitted by the laboratory along with the final data packages. Any changes to the analytical requests on the COC form or the laboratory purchase order (PO) shall be made in writing to the laboratory. A copy of this written change shall be sent to the project manager,

laboratory, and placed in the project files. The reason for the change shall be included in the sample log and project files so that recurring problems can be easily identified.

Following the completion of project activities, the sample logbooks, sample logs, and COC forms shall be sent to the Project Management Office (PMO) for storage in project files.

6.0 Training Requirements

Employees on projects generating waste must successfully complete the following training:

- Dangerous Goods Training
- Environmental Awareness Training
- Waste Management Training

7.0 Checklists

The HSE Self Assessment Checklist: Waste Analysis in Attachment 4 is provided as a method for verifying compliance with this SOP. The REM specifies the frequency in which this checklist shall be completed by the SC and provides this information in the project's written safety plan. The REM shall assist the SC in resolving any deficiencies identified during the self assessment. The REM may also use this checklist when performing HS audits at CH2M HILL projects, including subcontractor's activities.

8.0 References

- US Environmental Protection Agency (EPA): 40 CFR Parts 260 through 279, Resource Recovery and Conservation Act (RCRA)
- US Environmental Protection Agency (EPA): 40 CFR 761, Toxic Substances Control Act (TSCA,)
- US Department of Transportation (DOT): 49 CFR 171 through 180, Hazardous Materials

9.0 Attachments/ Appendices

Attachment 1: **Sample Label and Completion Instructions**

Attachment 2: **Custody Seal and Completion Instructions**

Attachment 3: **Chain of Custody Form and Completion Instructions**

Attachment 4: **Self-Assessment Checklist**

10.0 Approval

Revision	Date	Prepared By	Approved By:
1.0	10-08-2008	Jim Kelly	

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Attachment 1: Sample Label and Completion Instructions

Project Name: _____ Project No: _____

Sample ID: _____

Sample Date: _____ Sample Time: _____

Sampler(s): _____

Analyses: _____

Preservatives: _____

Completion Instructions:

The following information shall be recorded with a waterproof marker on each label:

- Project name
- Project number
- Sample identification or number
- Date and time of sample collection (24 hour clock)
- Sampler's name or initials
- Sample preservatives (if applicable)
- Analyses to be performed on the sample (specifically for the specific container and preservatives—typically for water samples only). This shall be identified by the method number (or name if the number is not known).

These labels may be obtained from the analytical laboratory or printed from a computer onto adhesive labels.

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Attachment 2: Custody Label and Completion Instructions

Signature: _____

Date and time_____ Time: _____

Completion Instructions:

The custody seals shall contain the following information:

- Signature
- Date and time the sample container (bottle/jar/vial) or sample shipping container (cooler) was sealed (24 hour clock)

The custody seals may be obtained from the analytical laboratory or printed from a computer onto adhesive labels.

Attachment 3: Chain of Custody Form and Instructions

Attachment 3: Chain of Custody Form and Instructions

Chain-of-Custody Form Instructions

The COC form completion procedures are crucial in properly transferring the custody and responsibility of samples from field personnel to the laboratory. This form also is important for accurately and concisely requesting analyses for each sample; it is essentially a release order from the analysis subcontract.

1. COC Number: Assign a unique ID that is linked to the project (e.g., 152901-001, GWM35-06-24- 99)
2. Project Name: Overall name of project (e.g., NAS Whiting Field)
3. Project Phase, Site, or Task: Name of specific site/task (e.g., Building 1429 excavation)
4. Project Contact: CH2M HILL contact person at site
5. Project Number: Project number assigned including WBS or charge code
6. Task Number: Task number assigned
7. Project Tel No. and Fax No.: Numbers where laboratory can call with questions or fax preliminary results
8. Laboratory Name and Contact: Name of lab where samples are to be sent and contact person
9. Lab PO Number: Laboratory Purchase Order number for the particular sampling event
10. Lab Tel No and Fax No: Numbers to call lab with questions and to fax copy of COC
11. Fax and Mail Reports to; Recipient 1 (Name and Company): Person and company where prelim and/or final copy of analytical results are to be sent
12. Fax and Mail Reports to; Recipient 2 (Name and Company): Person and company where prelim and/or final copy of analytical results are to be sent if to more than one
13. Fax and Mail Reports to; Recipient 3 (Name and Company): Person and company where prelim and/or final copy of analytical results are to be sent if to more than one
14. Recipient 1 (Address, Tel No., and Fax No.): Address, phone number and fax number of where prelim is to be faxed and final analytical results are to be sent
15. Recipient 2 (Address, Tel No., and Fax No.): Address, phone number and fax number of where prelim is to be faxed and final analytical results are to be sent if more than one

16. Recipient 3 (Address, Tel No., and Fax No.): Address, phone number and fax number of where prelim is to be faxed and final analytical results are to be sent if more than one

17. Item (numbered 1 through 10): to be used when relinquishing samples

18. Sample Identifier: Specific sample number for each sample NOT FOR EACH BOTTLE. For QC samples such as trip blanks, use TB-sample date-number (1, 2, 3...if more than one trip blank on that sample date).

19. Sample Description/Location: Describe sample and where it was collected (confirmation soil collected at NW wall of excavation A **or** groundwater sample collected at MW3 at OWS 13 **or** soil boring collected at 2' interval at OWS 13)

20. Matrix: Soil, Sediment, Water, Oil, Product, Vapor, Wipe, etc.

21. Date Collected: What day was sample collected

22. Time Collected: What time was sample collected (24-hour clock)

23. Data Package Level: CCI Data Package Level (A, B, or C)

24. TAT (Calendar Days): Place turn-around-time in days for when preliminary results are due

25. Analyses Required (Include Method Numbers): List one analysis per column and include method number (will be listed in Sampling and Analysis Plan) (TCL VOCs by 8260B)

26. Sample Type: Field, QC, Grab, Composite (include all applicable descriptions; e.g. Field Grab

27. Comments/Screening Readings: Place any comments here, for example, "strong organic odor." Also if soil was screened before sent to lab, place screening results here.

28. Lab ID: Leave this blank, this is for lab's use.

29. Sampler(s) and Company: ALL samplers' names and companies here.

30. Courier and Shipping Number: Put name of shipping company and airbill number(s) here. Include ALL airbill numbers. (Fed Ex #: 123456789, 987654321, 132435465)

31. Samples Temp and Conditions Upon Receipt: Leave blank, lab will fill in.

32. Relinquished by: Sign and print name and place date and time here. Date should be on or after date collected. (See the SOP for custodial instructions)

33. Received by: Sign and print name and place date and time here. (See the SOP for custodial instructions)

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Attachment 4: HSE Self-Assessment Checklist

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Attachment 4: HSE Self-Assessment Checklist

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HS&E Self-Assessment Checklist –Waste Management: Analysis and Characterization

This checklist shall be used by CH2M HILL personnel **only** and shall be completed at the frequency specified in the project's HSP/FSI. This checklist is to be used at locations where: (1) CH2M HILL employees will be managing wastes generated on project sites and/or (2) CH2M HILL provides oversight of subcontractor personnel who are managing wastes generated at project sites.

CH2M HILL staff shall not direct the means and methods of subcontractor waste management activities nor direct the details of appropriate corrective actions. The subcontractor must determine how to correct deficiencies and CH2M HILL staff must carefully rely on their expertise. Conditions considered to be imminently dangerous (possibility of serious injury or death) must be corrected immediately or all exposed personnel must be removed from the hazard until corrected.

The Safety Coordinator (SC) may consult with subcontractors when completing this checklist, but shall not direct the means and methods of waste characterization, sampling and analysis operations nor direct the details of corrective actions. Subcontractors shall determine how to correct deficiencies, and we must carefully rely on their expertise. Items considered to be imminently dangerous (possibility of serious injury or death) shall be corrected immediately or all exposed personnel shall be removed from the hazard until corrected.

Completed checklists shall be sent to the HS&E Staff for review.

Project Name: _____ Project No.: _____

Location: _____ PM:

Auditor: _____ Title: _____ Date: _____

This specific checklist has been completed to:

☐ Evaluate CH2M HILL compliance with its Waste Management: Analysis and Characterization program (SOP HSE-408)

☐ Evaluate a CH2M HILL subcontractor's compliance with its waste management program

Subcontractors Name: _____

- Check "Yes" if an assessment item is complete/correct
- Check "No" if an item is incomplete/deficient. Deficiencies shall be brought to the immediate attention of the subcontractor. Section 3 must be completed for all items checked "No."
- Check "N/A" if an item is not applicable
- Check "N/O" if an item is applicable but was not observed during the assessment

SECTION 1

	Yes	No	N/A	N/O
GENERAL WASTE CHARACTERIZATION INFORMATION (5.1)				
COMPLIANCE PROGRAM (5.1.1)				
1. Personnel told not to sign waste documentation (e.g., manifests) unless specifically authorized by the client in writing.	☐	☐	☐	☐
2. Waste characterized before it is generated.	☐	☐	☐	☐
3. Waste characterized by Client using generator information	☐	☐	☐	☐
4. Waste stream characterization documented in project file.	☐	☐	☐	☐
5. Waste volumes estimated.	☐	☐	☐	☐
6. Disposal facility sampling and analytical requirements identified.	☐	☐	☐	☐
WASTE SAMPLING AND ANALYSIS (5.2)				
IDENTIFY ANALYTICAL TEST METHODS (5.2.1)				
7. Nature and quantity of the waste determined.	☐	☐	☐	☐
8. Analyses required for transport, treatment and disposal determined.	☐	☐	☐	☐
9. Detection limits identified.	☐	☐	☐	☐
10. Disposal facility provided with analytical results.	☐	☐	☐	☐
11. Analytical test methods identified.	☐	☐	☐	☐

H&S Self-Assessment Checklist—Waste Management: Analysis and Characterization

	Yes	No	N/A	N/O
SAMPLING (5.3)				
12. A sampling plan is developed.	☐	☐	☐	☐
13. Field activities are recorded in a logbook.	☐	☐	☐	☐
14. Exceptions to sampling plan documented in field logbook.	☐	☐	☐	☐
Sample Labels (5.3.1)				
15. The sample is in custody.	☐	☐	☐	☐
16. Each container labeled with project name, number, sample ID number, date and time.	☐	☐	☐	☐
17. The label on the container is covered with clear tape to prevent loss.	☐	☐	☐	☐
Custody Seals (5.3.4)				
18. Sample shipping containers sealed with two custody seals.	☐	☐	☐	☐
19. Custody seals placed over the left and right sides of the container's cover (cooler).	☐	☐	☐	☐
20. Each seal signed and dated with time.	☐	☐	☐	☐
21. Seals are covered with clear tape to prevent loss.	☐	☐	☐	☐
22. Custody seals placed on sample container immediately after collection.	☐	☐	☐	☐
23. Custody seals must be placed in a manner that they must be broken to open sample container.				
CHANGE OF CUSTODY FORM INSTRUCTIONS (5.3.5)				
24. Chain of Custody form completed per instructions.	☐	☐	☐	☐
RECORDS (5.3.6)				
25. Official copy of COC form sent to the project chemist and lab with sample shipment.	☐	☐	☐	☐
26. Original COC submitted to the lab along with final data packages.	☐	☐	☐	☐
27. Changes to analytical requests on COC form or the PO made in writing to the lab.	☐	☐	☐	☐
28. A copy of written change sent to PM, lab and placed in project files.	☐	☐	☐	☐
29. Reasons for change are included in sample log and project file.	☐	☐	☐	☐
30. Sample logbooks, sample logs, and COC forms sent to PM at completion of project activities.	☐	☐	☐	☐

SECTION 3

Complete this section for all items checked "No' in Sections 1 or 2. Deficient items must be corrected in a timely manner.

Item #	Corrective Action Planned/Taken	Date Corrected

Auditor: _____ Project Manager: _____

CLICK HERE FOR ATTACHMENTS

Waste Management: Non-Hazardous Waste
Enterprise Standard Operating Procedure HSE-411

1.0 Purpose

This Enterprise Standard Operating Procedure (SOP) describes the management practices associated with non-hazardous waste. Waste management includes storage, transportation, and disposal of waste. It is designed to be used in conjunction with HSE-413, Waste Management Planning and HSE-408, Waste Management Analysis and Characterization and applies to field projects as well as office and warehouse wastes.

2.0 Scope and Application

2.1 Scope

Non-hazardous wastes are generally subject to solid waste management requirements. Other wastes that are not hazardous but are subject to regulation include asbestos waste (see HSE-502), PCB waste (see HSE-412), and lead-contaminated waste (see HSE-508). However, non-hazardous waste may be more stringently regulated by a state or local entity. Consult the Responsible Environmental Manger to determine state or local management standards.

2.2 Application

This SOP applies Enterprise Wide to all CH2M HILL Legal Entities and Business Groups, their employees, subcontractors and their lower-tier subcontractors that operate in the United States (US) and internationally.

Some state environmental or OSHA programs may have more stringent requirements. . State regulations may be found under Regtools. Contact the appropriate Responsible Environmental Manager (REM) to address these specific requirements. This SOP should be used as a starting point for international operations, but country-specific health, safety and environmental (HSE) regulations shall prevail, and a country-specific SOP should be developed to comply with these specific HSE regulations.

2.3 Applicable Enterprise SOPs

Applicable Enterprise Standards of Practice and Standard Operating Procedures that are applicable to this Waste Management-Non Hazardous Waste SOP are as follows:

- HSE-408, Waste Management Analysis and Characterization
- HSE-409, Waste Management
- HSE-412, PCB Waste
- HSE-413, Waste Management Planning
- HSE-415, Universal Waste
- HSE-502, Asbestos Waste
- HSE-508, Lead-contaminated Waste

3.0 Definitions

3.1 Asbestos-Containing Material (ACM)

Asbestos-containing materials are materials that contain asbestos that may be generated from construction, demolition or renovation of structures where asbestos is present; or removal of materials containing asbestos.

3.2 Characterization

Characterization is the process performed by a generator to determine the type of waste being managed and how the waste should be disposed. This process can involve identifying waste constituent concentrations and comparing the concentrations to legal requirements or threshold levels that affect how the waste is disposed. Waste characterization procedures are provided in the HSE-408, Waste Management Analysis and Characterization.

3.3 Construction and Demolition (C&D) Debris

Construction and demolition (C&D) debris is waste material that is produced in the process of construction, renovation or demolition of structures. Structures can include buildings, bridges, slabs, and pavements. Components of C&D debris typically include concrete, asphalt, wood, metals, gypsum wallboard, and roofing. Note that some states have separate regulatory requirements for C&D waste.

3.4 Lead-contaminated Waste

Lead-contaminated wastes are wastes that contain lead (e.g., old paint containing lead). Such wastes may be generated during construction, demolition, or renovation of structures. HSE-508, Lead-contaminated Waste, discusses managing lead-contaminated materials

3.5 Non-Hazardous Waste

In this SOP, non-hazardous wastes are considered to be wastes that are not classified as hazardous under RCRA Subtitle C and are not subject to other regulatory requirements (e.g., PCB, asbestos).

3.6 National Pollutant Discharge Elimination System (NPDES)

The National Pollutant Discharge Elimination System (NPDES) permit program regulates the point source discharge of industrial wastewater and storm water.

3.7 Publicly Owned Treatment Works (POTW)

Publicly-owned treatment works (POTW), owned by a state or municipality, are used to treat (including recycle and reclaim) either domestic sewage or a combination of domestic sewage and industrial waste of a liquid nature. If owned by a federal agency (e.g., Department of Defense), this is called a federally-owned treatment works (FOTW).

3.8 Purge Water

Purge water is removed from a well prior to the collection of a groundwater sample to make sure that the sample is representative of groundwater in the aquifer and not stagnant water within the well.

3.9 Petroleum-Contaminated Soil

Petroleum-contaminated soil refers to soil that may be contaminated with petroleum as a result of leaky underground storage tanks or some other source.

3.10 PCB Wastes

Most PCB wastes that are encountered are generated from management of PCB-containing equipment (e.g., light ballasts), clean up and remediation of PCB-contaminated soils. This also includes PCB articles—any PCB item, PCB container, PCB equipment, or anything that deliberately or unintentionally contains PCBs.

3.11 Solid Waste

For purposes of this SOP, solid wastes are generally non-hazardous wastes that do not contain any regulated constituents such as solvents, lead, and PCBs.

3.12 Stockpiles

A stockpile is an area where soil or debris is consolidated and stored or managed in a pile.

3.13 Underground Injection Control (UIC)

The Underground Injection Control (UIC) Program refers to the Environmental Protection Agency (EPA) program responsible for monitoring subsurface wastewater discharges.

3.14 Universal Waste

Universal waste is hazardous waste that is regulated by EPA or state jurisdictions having authority over hazardous waste regulation. If managed as universal waste, these hazardous wastes are subject to less stringent management procedures. These wastes currently include batteries, agricultural pesticides, mercury-containing thermostats, and lamps.

3.15 Well Development Water

Well development waster refers to water that is circulated through the well screen and well bore to remove settled and suspended materials within the well, which increases the permeability of the formation surrounding the well screen.

4.0 Roles and Responsibilities

4.1 Project Manager (PM)

The PM is responsible for ensuring that non-hazardous wastes are managed in accordance with this SOP. The PM should work with the Responsible Environmental Manager (REM) to identify good management practices and comply with any local requirements affecting non-hazardous or solid wastes.

4.2 Responsible Environmental Manger (REM)

The REM assists the project manager in ensuring that the project complies with the environmental laws and regulations by identifying issues and resources to meet project needs.

4.3 Safety Coordinator (SC)

The SC is familiar with project plans, including air emission controls and permit conditions, and implements the plans in the field.

5.0 Procedures

CH2M HILL requires managing non-hazardous wastes in accordance with good management practices.

5.1 Waste Characterization and Management

To assure proper management and disposal, it is necessary to determine the type of waste that needs to be managed. Refer to HSE-408, Waste Management Analysis and Characterization for waste characterization procedures. The following wastes are subject to other requirements that are discussed in the corresponding SOP:

- **Asbestos—**Refer to HSE-502. Asbestos-containing wastes must be double-bagged and placed in landfill cells permitted to accept asbestos-containing wastes.
- **PCBs—**Refer to HSE-412.
- **Universal Waste—**Refer to HSE-415. If universal waste is not managed according to the universal waste regulatory requirements, it is considered hazardous waste and would need to be managed as discussed in HSE-409.

Some states and local agencies require that petroleum-contaminated soil be managed in a more stringent manner, and may even require that it be managed as hazardous waste. Consult the REM for state and local requirements.

For all waste streams, CH2M HILL's order of preference for waste management is source reduction, recycling, and lastly disposal.

5.1.1 Source Reduction

Source reduction is defined as the design, manufacture, and use of products to reduce the quantity and toxicity of waste produced when the products reach the end of their useful lives. Source reduction activities fall into some basic categories:

- Product reuse (e.g., reusable shopping bags and coffee mugs)
- Reduced material volume (e.g., less unnecessary packaging for products)
- Reduced toxicity of products (e.g., use substitutes for lead, cadmium, mercury, and other toxins)
- Increased product lifetime (e.g., design products with longer useful life)
- Decreased consumption (e.g., changing consumer buying practices, bulk purchasing).

These source reduction practices reduce cost and amount of waste management practices.

5.1.2 Recycling

A variety of office and project wastes can be recycled. Check with your REM to see what markets are available in your area.

5.1.3 Office Recycling

The following materials are recycled at CH2M HILL offices:

- Aluminum cans
- Corrugated cardboard
- Laser printer cartridges
- Newspaper
- Office paper
- Magazines
- Telephone books
- Lead-acid batteries
- Selected plastics

5.1.4 Project Recycling

In addition to the office recyclables that are generated on project sites, the following project materials should also be recycled:

- Asphalt
- Concrete
- Insulation
- Scrap metal
- Petroleum-contaminated soil
- Carbon filters

5.2 Storage

The storage of solid waste (including items that may be recycled, e.g., petroleum-contaminated soil) may be subject to state or local requirements. Consult your REM for assistance.

5.2.1 Stockpiles

Solid waste stockpiles are typically regulated by state and local agencies. Technical standards may include storing the wastes on plastic sheeting and covering with plastic. Some states require permits for solid waste stockpiles. States may also have time limits for solid waste stockpiles and dust control requirements. Consult your REM for assistance.

5.2.2 Containers

Solid waste containers should meet DOT specifications. State and local agencies may also have specific requirements for solid waste containers. Consult your REM for assistance.

5.2.3 Labels

Use the following label for containers of non-hazardous waste. The non-hazardous waste label and other labels can be ordered by phone or online through Mesa Label Express (www.mesalabel.com; 858-693-4987) or Labelmaster (www.labelmaster.com; 800-621-5808).

5.3 Disposal

Non-hazardous wastes are subject to regulation as solid waste. Consult your REM for assistance. The following subsections discuss management of typical non-hazardous wastes generated at CH2M HILL project sites.

5.3.1 Construction and Demolition (C&D) Debris

Demolition debris can be hauled, and then dispensed at any construction and demolition (C&D) landfill or any municipal solid waste landfill that is permitted to accept C&D debris. C&D debris should be reduced whenever possible. Clean C&D debris can be reused, such as broken concrete without protruding metal bars, bricks, rock, stone, reclaimed asphalt pavement, dirt or sand. Additionally, there are a number of C&D debris recyclers that will accept wood, aluminum and other metals, asphalt, concrete, and corrugated cardboard. If debris is known to contain hazardous substances, review HSE-413, Waste Management Planning and HSE-408, Waste Management Analysis and Characterization to determine the appropriate procedures for disposal.

5.3.2 Lead-Contaminated Waste

If lead-based paint debris from abatement, deleading, renovation, remodeling, and demolition contains more than 5 mg/L of lead (determined by knowledge of the waste or TCLP tests), the debris may be subject to RCRA subtitle C hazardous waste requirements. For RCRA requirements and procedures, refer to HSE-413, Waste Management Planning and HSE-408, Waste Management Analysis and Characterization. EPA has proposed a rule to exclude lead-based paint from regulation as hazardous waste, although it is regulated under the Toxic Substances Control Act (TSCA).

5.3.3 Petroleum-Contaminated Soil

Petroleum-contaminated soil is generated from spilled petroleum compounds, often from leaky underground storage tanks. The soil can be treated, disposed of, recycled, and reused in many different manners. Typical strategies that have been used to manage petroleum-contaminated soil include: use of the petroleum-contaminated soil as a landfill cap, removal of the petroleum through soil treatment with Low-Temperature Thermal Desorption (LTTD), and soil land farming to break down the petroleum products with reuse onsite.

LTTD has proven very effective in reducing concentrations of petroleum products including gasoline, jet fuels, kerosene, diesel fuel, heating oils, and lubricating oils. Operation of LTTD units at a project site requires various permits and demonstration of compliance with permit requirements. Monitoring of LTTD system waste streams (*e.g.*, concentrations of particulates, volatiles, and carbon monoxide in stack gas) are required by the agency(ies) issuing the permits for operation of the facility. Each state has its own standards for soil cleanup levels. Contact your REM for assistance as soil and air monitoring may be required.

5.3.4 Well Purge and Development Water

There are four options for the disposal of well purge and development water generated at project sites:

1. Onsite treatment and discharge
2. Discharge to POTW/FOTW
3. Discharge to injection galleries/injection wells
4. Discharge to ground

Note that discharge to surface water is not an option. A discharge to surface water would require an NPDES permit.

Onsite Treatment and Discharge

Non-hazardous well purge/development water may be treated with an existing groundwater treatment system that has an NPDES permit or POTW approval. It can also be discharged to the sewer without treatment if the POTW grants approval and the POTW's influent criteria are met. Purge water can also be discharged to an onsite wastewater pretreatment system operated by the facility owner, providing it does not affect the discharge from the pretreatment system.

Hazardous purge/well development water can also be treated onsite in a treatment system without a hazardous waste treatment permit if the treatment system can be defined as a wastewater treatment unit under RCRA (40 CFR 264.1 (g)(10)). To do so, the following criteria must be met:

1. The treatment unit has an NPDES permit under the Clean Water Act (CWA), or discharges to a POTW via sewer or direct connection, under a permit or written approval;

2. The treatment unit receives and treats (or stores) influent wastewater, which might be hazardous; and

3. The treatment unit meets the definition of a *tank* or *tank system*.

If discharged to a POTW, the water is no longer defined as a "solid waste" under RCRA once it is in the sewer because it is regulated under the CWA.

In addition to the above, the water should originate from wells at the site and not from other sites; i.e., from the same aquifer. Do not treat water from offsite sources unless instructed by the client in writing, and only after receiving EPA or state approval.

Although treatment devices, such as carbon canisters, air strippers, and oil/water separators are often considered a tank or tank system, these definitions are subject to state and local

regulatory interpretation. Waste residues, e.g., filter bags, and tower packing, from water treatment are not exempt from the RCRA regulations and must be tested to determine if they are a hazardous waste.

Discharge at Offsite POTW/FOTW

Hazardous and non-hazardous well purge/development water may be discharged at an offsite POTW under the following conditions:

1. The discharged water is properly classified, using "client" knowledge and/or testing;
2. The water must *not* be ignitable and must not have a sheen or film of separate-phase oil or other organics;
3. A written notice, signed by the client, is submitted to the POTW, and the water meets the POTW acceptance limits (the client determines this);
4. Approval from the POTW is received *in writing*; and
5. For hazardous waste shipments to POTWs, federal regulations in 40 CFR 270.1(c)(2)(v) also require the POTW to:

    - Have an EPA ID number;
    - Request use of a Hazardous Waste Manifest for the shipment;
    - Report manifest discrepancies;
    - Submit Biennial and unmanifested waste reports; and
    - The waste must meet pre-treatment requirements.

This is not a preferred option. It is also impractical because it is difficult to find a POTW that will accept RCRA or state hazardous wastes. If a POTW is used, document the shipment with a shipping paper or manifest, and make arrangements for transportation with a transporter approved for use by CH2M HILL and the client.

Discharge to Injection Galleries/Injection Wells

Discharge of well purge/development water to an injection gallery or injection well is allowed by permit or approval. The purge water must originate from wells at the site, i.e., from the same aquifer, and have the same chemical properties as that approved for re-injection. The criteria below must be met:

1. The water is non-hazardous or is exempt from hazardous waste regulations under 40 CFR 261.4(a) &(b); and
2. The injection gallery at the client site is operating under a state permit, or approval from the EPA Regional Underground Injection Control (UIC) Coordinator; or
3. For CERCLA Sites, if the water is hazardous and is discharged to an injection gallery or injection well constructed within a designated area of contamination, the agency will likely establish discharge limits, and monitoring/reporting requirements through the state UIC or wastewater discharge program.

Discharge to Ground Surface

Discharge of purge water to ground surface is allowable under the following conditions:

1. The discharged water is properly classified, using client knowledge and/or testing;
2. The water is *not* a RCRA or state hazardous waste;
3. Written approval is received from the client; and
4. Discharge is performed under state permit or approval, or state guidelines.

States often require that purge water be filtered with carbon prior to discharge to ground surface. Portable handcart-mounted adsorption units are available from a number of vendors such as Continental Environmental Systems (800-342-1103). Remember that a container of granular activated carbon (GAC) that shows signs of "breakthrough" is considered "spent" and must be managed as a waste and must be characterized under procedures discussed in the Waste Management Analysis and Characterization SOP (HSE-408).

6.0 Training Requirements

Employees on projects that generate waste must complete the following training:

- Dangerous Goods Training
- Environmental Awareness Training
- Waste Management Training

7.0 Checklists

The HSE Self Assessment Checklist: Non Hazardous Waste Management in Attachment 1 is provided as a method for verifying compliance with this SOP. The REM specifies the frequency in which this checklist shall be completed by the SC and provides this information in the project's written safety plan. The REM shall assist the SC in resolving any deficiencies identified during the self assessment. The REM may also use this checklist when performing HS audits at CH2M HILL projects, including subcontractor's activities.

8.0 References

The following regulations were referenced to prepare this Enterprise Standard Operating Procedure:

- US Environmental Protection Agency (EPA): 40 CFR Parts 260 through 279, Resource Recovery and Conservation Act (RCRA)

9.0 Attachments

Attachment 1: **HS&E Self Assessment Checklist: Non-Hazardous Waste**

10.0 Approval

Revision	Date	Prepared By	Approved By:
1.0	10-08-2007	Jim Kelly	

CH2MHILL

Attachment 1: HSE Self-Assessment Checklist

CH2MHILL

HS&E Self-Assessment Checklist –Non-Hazardous Waste

This checklist shall be used by CH2M HILL personnel **only** and shall be completed at the frequency specified in the project's HSP/FSI. This checklist is to be used at locations where CH2M HILL generate or manage non-hazardous waste.

CH2M HILL staff shall not direct the means and methods of subcontractor activities nor direct the details of appropriate corrective actions. The subcontractor must determine how to correct deficiencies and CH2M HILL staff must carefully rely on their expertise. Conditions considered to be imminently dangerous (possibility of serious injury or death) must be corrected immediately or all exposed personnel must be removed from the hazard until corrected.

Project Name: _____ Project No.: _____

Location: _____ PM:

Auditor: _____ Title: _____ Date: _____

This specific checklist has been completed to:

☐ Evaluate CH2M HILL compliance with its Non-Hazardous Waste SOP (HSE-411)

☐ Evaluate a CH2M HILL subcontractor's compliance with non-hazardous waste procedures.

Subcontractors Name: _____

- Check "Yes" if an assessment item is complete/correct
- Check "No" if an item is incomplete/deficient. Deficiencies shall be brought to the immediate attention of the subcontractor. Section 3 must be completed for all items checked "No."
- Check "N/A" if an item is not applicable
- Check "N/O" if an item is applicable but was not observed during the assessment

SECTION 1

	Yes	No	N/A	N/O
PROCEDURES				
SOURCE REDUCTION AND RECYLCING (5.1.1)				
1. Products have been re-used to reduce waste quantity and toxicity	☐	☐	☐	☐
2. Material volumes have been reduced by less packaging.	☐	☐	☐	☐
3. Less toxic products have been used to reduce waste toxicity.	☐	☐	☐	☐
4. Materials at CH2M HILL offices are recycled.	☐	☐	☐	☐
5. Recyclables generated at project sites are recycled.	☐	☐	☐	☐
STORAGE (5.2)				
6. Local or state solid waste storage requirements have been identified.	☐	☐	☐	☐
7. Local and state solid waste stockpile requirements have been identified.	☐	☐	☐	☐
8. Solid waste containers meet DOT specifications.	☐	☐	☐	☐
9. Non-hazardous waste label used for containers of non-hazardous waste.	☐	☐	☐	☐
DISPOSAL (5.3)				
Construction and Demolition Debris (5.3.1)				
10. Construction debris is disposed of at a landfill permitted to take C&D debris.	☐	☐	☐	☐
11. Clean C&D debris is reused.	☐	☐	☐	☐
12. C&D debris considered for recycling.	☐	☐	☐	☐
13. C&D debris containing hazardous waste is managed under HSE-408.	☐	☐	☐	☐

	Yes	No	N/A	N/O
Lead-Contaminated Waste (5.3.2)				
14. Lead-based paint debris managed under HSE-408 and HSE-413.	☐	☐	☐	☐
Petroleum-Contaminated Soil (5.3.3)				
15. REM consulted for treatment, disposal and recycling options for petroleum-contaminated soils.	☐	☐	☐	☐
Well Water Onsite Treatment and Discharge				
16. Non-hazardous well purge/development water treated in existing NPDES-permitted system.	☐	☐	☐	☐
17. Non-hazardous water is discharged to sewer untreated with POTW approval.	☐	☐	☐	☐
18. Non-hazardous waste is discharged to onsite wastewater pretreatment system.	☐	☐	☐	☐
19. Treatment of hazardous wastewater meets requirements of RCRA wastewater treatment unit.	☐	☐	☐	☐
Discharge at Offsite POTW/FOTW (5.3.4)				
20. Discharged water is classified using "client" knowledge and/or testing.	☐	☐	☐	☐
21. The water is not ignitable, does not contain organics or have an oily sheen.	☐	☐	☐	☐
22. Client provided written notice to POTW that water meets acceptance limits.	☐	☐	☐	☐
23. POTW discharge approval received in writing.	☐	☐	☐	☐
24. POTW EPA ID number obtained for hazardous wastewater.	☐	☐	☐	☐
25. Hazardous waste manifest used for transport of hazardous waste to POTW.	☐	☐	☐	☐
26. Waste meets pre-treatment requirements	☐	☐	☐	☐
Discharge to Injection Galleries/Injection Wells (5.3.4)				
27. Permit or approval obtained for discharge to injection gallery or well.	☐	☐	☐	☐
28. Purge water originated from wells at the site (same aquifer with same chemical properties).	☐	☐	☐	☐
29. The purge water is non-hazardous or exempt from hazardous waste regulations.	☐	☐	☐	☐
30. Injection gallery at site is operating under state permit or approval from EPA.	☐	☐	☐	☐
Discharge to Ground Surface (5.3.4)				
31. Discharged water is classified using "client" knowledge and/or testing.	☐	☐	☐	☐
32. Purge water is not a RCRA or state hazardous waste.	☐	☐	☐	☐
33. Written approval received from the client.	☐	☐	☐	☐
34. State permit or approval received for discharge.	☐	☐	☐	☐
35. Carbon filtration used prior to discharge to the ground surface.	☐	☐	☐	☐

SECTION 3

Complete this section for all items checked "No' in Sections 1 or 2. Deficient items must be corrected in a timely manner.

Item #	Corrective Action Planned/Taken	Date Corrected

Auditor: _____ Project Manager: _____

CH2MHILL

CLICK HERE FOR ATTACHMENTS

Waste Management: PCBs
Enterprise Standard Operating Procedure HSE-412

1.0 Purpose

This Enterprise Standard Operating Procedure (SOP) describes the procedures necessary for proper management of wastes containing polychlorinated biphenyls (PCBs). PCB wastes may be generated from cleanup and remediation of PCB-contaminated soils or from PCB articles removed from service.

2.0 Scope and Application

2.1 Scope

PCBs are regulated under the Toxic Substances Control Act (TSCA) in the United States. Unlike the hazardous waste regulations, the states are not responsible for implementing the laws governing management of materials containing PCBs over 50 ppm. Note that states may regulate PCBs between 1 and 50 ppm; usually these lower PCB concentrations must be managed as a hazardous waste.

2.2 Application

This SOP applies Enterprise-Wide to all CH2M HILL Legal Entities and Business Groups, their employees, subcontractors and their lower-tier subcontractors that operate in the United States (US) and internationally.

Some state environmental or OSHA programs may have more stringent requirements. State regulations may be found under Regtools. Contact the appropriate Responsible Environmental Manager (REM) to address these specific requirements. This SOP should be used as a starting point for international operations, but country-specific health, safety and environmental (HSE) regulations shall prevail, and a country-specific SOP should be developed to comply with these specific HSE regulations.

Subcontractors who handle universal waste are responsible for managing them in accordance with applicable regulations and their own safety procedures.

2.3 Applicable Enterprise SOPs

Applicable Enterprise Standards of Practice and Standard Operating Procedures that are applicable to this PCBs SOP are as follows:

- HSE-305, **Demolition SOP**
- HSE 403, **Hazardous Materials Handling**
- HSE-407, **Stockpiles**
- HSE-409, **Waste Management: Hazardous Waste**
- HSE-415, **Waste Management: Universal Waste**

3.0 Definitions

3.1 Polychlorinated Biphenyls (PCBs)

PCB and PCBs means any chemical substance(s) that is limited to the biphenyl molecule that has been chlorinated to varying degrees or any combination of substances that contain such substance. PCBs were used in hundreds of industrial and commercial applications including electrical, heat transfer, and hydraulic equipment; as plasticizers in paints, plastics and rubber products; in pigments, dyes, and carbonless copy paper; and many other applications.

3.2 Waste Containing PCBs

Waste containing PCBs is used in this SOP to include any waste stream that includes any concentration of PCBs. This is different from the U.S. term "PCB waste," which has a more specific regulatory definition.

3.3 PCB Articles

PCB article means any manufactured article, other than a PCB container, that contains PCBs and whose surface(s) has been in direct contact with PCBs.

3.4 PCB Bulk Product Waste

PCB bulk product waste means waste derived from manufactured products containing PCBs in a non-liquid state, at any concentration where the concentration at the time of designation for disposal was ≥50 ppm PCBs. PCB bulk product waste includes:

- Non-liquid bulk wastes or debris from the demolition of buildings and other man-made structures manufactured, coated, or serviced with PCBs. PCB bulk product waste does not include debris from the demolition of buildings or other man-made structures that is contaminated by spills from regulated PCBs (these are regulated as remediation waste).
- PCB-containing wastes from the shredding of automobiles, household appliances, or industrial appliances.
- Plastics (such as plastic insulation from wire or cable; radio, television and computer casings; vehicle parts; or furniture laminates); preformed or molded rubber parts and components; applied dried paints, varnishes, waxes or other similar coatings or sealants; caulking; adhesives; paper; Galbestos; sound-deadening or other types of insulation; and felt or fabric products, such as gaskets.
- Fluorescent light ballasts containing PCBs in the potting material.

3.5 PCB Remediation Waste

- PCB remediation waste means waste containing PCBs as a result of a spill, release, or other unauthorized disposal at concentrations ≥50 ppm PCBs, regardless of the concentration of the original spill; and materials which are currently at any concentration where the original source was ≥500 ppm PCB. PCB remediation waste means soil, rags, and other debris generated as a result of any PCB spill cleanup, including:

- Environmental media containing PCBs, such as soil and gravel; dredged materials, such as sediments, settled sediment fines, and aqueous decantate from sediment.
- PCB sewage sludge containing ≥500 ppm PCB; commercial or industrial sludge contaminated as the result of a spill of PCBs including sludges located in or removed from any pollution control device; aqueous decantate from an industrial sludge.
- Buildings and other man-made structures, such as concrete or wood floors or walls contaminated from a leaking PCB or PCB-contaminated transformer, porous surfaces and non-porous surfaces.

3.6 PCB-Contaminated Electrical Equipment

PCB-contaminated electrical equipment means any electrical equipment, including transformers, capacitors, circuit breakers, reclosers, voltage regulators, switches, electromagnets, and cable, that contains PCBs at concentrations of ≥50 ppm.

3.7 Radioactive PCB Waste

PCB/radioactive waste includes PCBs regulated for disposal that also contain source, special nuclear, or byproduct material that is subject to the Atomic Energy Act (AEA), or naturally occurring or accelerator-produced radioactive material.

4.0 Roles and Responsibilities

4.1 Project Manager

The Project Manager (PM) is responsible for ensuring the project is implemented in compliance with environmental requirements. The PM or his designee should work with the Responsible Environmental Manager to identify CH2M HILL responsibilities, costs and plan implementation.

4.2 Responsible Environmental Manager (REM)

The REM assists the project manger in ensuring that the project complies with environmental laws and regulations by identifying issues and resources to meet project needs.

4.3 Safety Coordinator (SC)

The SC is responsible for assisting the PM in storing, transporting, and disposing of PCB waste generated in the field in accordance with this SOP.

5.0 Procedures

CH2M HILL requires the management of wastes in compliance with applicable legal requirements.

5.1 Storage and Transport

5.1.1 Storage Limits

Wastes containing PCBs should generally be stored for no longer than 30 days to avoid more stringent storage and notification requirements.

5.1.2 Storage for Less than 30 Days

Wastes containing PCBs ≥50 ppm may be stored for 30 days without notification to EPA or a state agency, if:

- Containers or tanks are non-leaking
- A Spill Prevention Countermeasure and Control (SPCC) plan is in place if liquids are present
- Liquid PCB waste is managed in DOT-approved drums or stationary bulk storage tanks
- The generic identification number, "40 CFR PART 761" is written on manifests, records and reports if the facility does not have an EPA ID number

5.1.3 Storage Greater than 30 Days

Storing PCB wastes for more than 30 days requires an EPA identification number, and the storage area must meet more stringent design requirements. Storage area requirements (40 CFR 761.65(b)) for storing PCBs greater than 30 days include:

- Adequate roof and walls to keep rainfall from the PCB items
- 6-inch continuous curb around the items with a containment volume equal to/greater than twice the largest PCB item
- Containment area constructed of cement or non-porous material
- No drains, expansion joints or other openings which could allow liquid to escape
- Area not located within a 100-year floodplain

PCB equipment and containers can be stored in a storage area meeting specific requirements for up to 1 year. PCB/radioactive wastes are not subject to the 1-year storage limit as long as disposal efforts under AEA are documented. Bulk PCB remediation waste or PCB bulk product waste can be stored at the cleanup site for 180 days as long as covers, liners, etc., are provided [see 40 CFR 761.65(c)(9)].

The storage limit of PCB articles designated for reuse is five years. Resource Conservation and Recovery Act (RCRA) facilities may store TSCA-regulated PCB wastes without a separate TSCA permit. This gives generators more options when shipping TSCA wastes off-site, because small quantities of PCB waste can be sent to a RCRA facility for temporary storage, rather than having to send to a TSCA-permitted facility which may be further away and cost more.

TIP: Since it normally takes 3-6 months for a disposal facility to arrange transport, plan waste pickup well in advance of waste generation.

5.2 Storage Options

DOT container/packaging requirements apply to PCB storage. Refer to HSE-401, Hazardous Materials Handling or consult your REM.

Alternative storage methods may be available but must be approved by EPA as risk-based storage (PCB remediation waste or PCB bulk product waste) or granted a TSCA PCB Coordinated Approval. Consult the REM for assistance with alternative storage methods.

5.3 Label

PCB wastes regulated under TSCA (concentrations > 50 ppm) must be marked and labeled using the following EPA-approved label:

Note that some states also have labeling requirements for wastes containing low concentrations of PCBs (e.g., a blue label similar to the yellow PCB label).

5.4 Manifest

PCB waste regulated under TSCA must be transported with a manifest, using a uniform hazardous waste manifest (EPA Form 8700-22). The manifest must state the serial or other identifying number of the PCB waste (e.g., if the waste is a transformer), the date of removal from service, and the weight in kilograms of the PCB waste in each transformer. Refer to HSE-409: Waste Management: Hazardous Waste SOP for manifest procedures.

5.5 Transport

PCB wastes must be marked, labeled and transported in compliance with DOT requirements. Refer to HSE 407, Stockpiles or the regional DOT expert for more information.

5.6 Disposal Procedures

5.6.1 Determination of PCB Content

Disposal options are determined by the type of PCB item and PCB concentration. Determine PCB concentration using one of the following options: (1) sample the waste to determine concentration and apply the regulations specified for the type of material, or (2) assume the concentration to be 500 parts per million (ppm) or greater. Under the second option, it is not necessary to determine the PCB concentration of the material, but the most restrictive regulatory requirements apply.

The PCB disposal requirements are applied to the individual phase with the highest PCB concentration. Alternatively, phases may be separated and disposed of using the PCB disposal requirements that apply to each separated, single-phase material. Do not avoid disposal procedures by diluting the PCBs.

PCBs can be found in liquid, non-liquid and multi-phasic (combinations of liquid and non-liquid) forms. Use the following criteria to determine PCB concentrations:

- PCB concentrations for non-liquid PCBs should be based on a dry weight basis.
- PCB concentrations for liquid PCBs should be based on a wet weight basis. Liquid PCBs containing more than 0.5 percent by weight non-dissolved material shall be analyzed as multi-phasic non-liquid/liquid mixtures in which the phases shall be separated before chemical analysis.

5.6.2 Disposal of PCB Waste < 50 ppm

PCB waste containing less than 50 ppm of PCBs is not regulated under TSCA. However, disposal may be regulated by the state where the project is located. Consult the regional REM to determine state requirements.

5.6.3 Bulk Product Waste

There are four options for the disposal of PCB bulk product wastes: (1) performance-based disposal (incineration or chemical waste landfill); (2) disposal in solid waste landfills; (3) risk-based disposal (an EPA case-by-case basis); and (4) disposal as daily landfill cover or roadbed. The performance-based option includes several disposal options, such as disposal in RCRA Subtitle D landfills, TSCA chemical waste landfills, and thermal decontamination. Bulk waste may be disposed of in solid waste landfills, if it meets certain criteria (i.e., if the PCBs are tightly bound in the matrix of the waste and leach <10 μg/liter; or if the waste is automobile and appliance shredder fluff that does not contain PCB small capacitors). Under the risk-based option, EPA will evaluate applications for storage and disposal of PCB bulk product wastes on a case-by-case basis to determine whether a proposed method would result in unreasonable risks to human health.

5.6.4 PCB Small Capacitors

PCB small capacitors (e.g., fluorescent light ballasts) can be disposed in municipal landfills [40 CFR 761.60 (b)(2)(ii)]. These wastes are often encountered during demolition projects. More specific requirements for fluorescent light ballasts and other demolition wastes are found in HSE-305, Demolition and HSE-415, Waste Management: Universal Waste.

5.6.5 Remediation Waste

There are three options for disposal of remediation waste. These include the use of (1) the self-implementing disposal; (2) performance-based disposal; and (3) the risk-based disposal options.

Self-Implementing Cleanup

The self-implementing cleanup level (i.e., the ``walk-away'' level) for soil in high-occupancy (e.g., residential) areas is ≤1 ppm, or ≤10 ppm if the soil is capped. The cleanup level in low-occupancy (e.g., electrical substation) areas is ≤25 ppm to ≤100 ppm, depending on site conditions.

Decontamination standards for surfaces are as follows: for non-porous surfaces in contact with liquid PCBs destined for reuse, ≤10 micrograms PCBs per 100 square centimeters (10 □g PCBs/100 cm^2); for non-porous surfaces in contact with liquid PCBs destined for smelting, <100 □g PCBs/100 cm^2; for non-porous surfaces in contact with non-liquid PCBs destined for reuse, Visual standard No. 2, Near-White Blast Cleaned Surface Finish, of the National Association of Corrosion Engineers (NACE); for non-porous surfaces in contact with non-liquid PCBs destined for smelting, Visual standard No. 3, Commercial Blast Cleaned Surface Finish, of NACE; and for fresh spills to concrete, ≤10 □g PCBs/100 cm^2.

Decontamination standards for liquids are as follows: for water, <0.5 □g PCBs/L (approximately 0.5 ppb) for unrestricted use; and for organic and non-aqueous inorganic liquids, <2 □g PCBs/L (approximately 2 ppm).

EPA must be notified at least 30 days prior to the clean up. Consult the regional ECC for notification procedures. EPA must respond in writing within 30 days.

Cleanup levels are based on the kind of material and the potential exposure to PCBs left after cleanup is completed.

- The cleanup level for bulk PCB remediation waste in high occupancy areas is 1 ppm without further conditions. High-occupancy areas where bulk PCB remediation waste remains at concentrations between 1 ppm and 10 ppm shall be covered with a cap meeting EPA requirements.
- The cleanup level for bulk PCB remediation waste in low-occupancy areas is 25 ppm unless otherwise specified in this paragraph. Bulk PCB remediation wastes may remain at a cleanup site at concentrations between 25 ppm and 50 ppm if the site is secured by a fence and marked with a sign including the PCB label. Bulk PCB remediation wastes may remain at a cleanup site at concentrations between 25 ppm and 100 ppm if the site is covered with a cap meeting EPA requirements (note that the state may have other requirements).
- Any person cleaning up bulk PCB remediation waste on-site using a soil washing process may do so without EPA approval, subject to all of the following
- A non-chlorinated solvent is used.
- The process occurs at ambient temperature.
- The process is not exothermic.
- The process uses no external heat.
- The process has secondary containment to prevent any solvent from being released to the underlying or surrounding soils or surface waters.
- Solvent disposal, recovery, and/or reuse is in accordance with applicable EPA requirements.

Off-Site Disposal

Bulk PCB remediation waste may be sent off-site for decontamination provided the waste is either dewatered on-site or transported off-site in containers meeting DOT requirements. Any person disposing off-site of dewatered bulk PCB remediation waste shall do so as follows: Bulk PCB remediation wastes with a PCB concentration 50 ppm shall be disposed of in a hazardous waste landfill permitted by EPA under RCRA, or by a state authorized under RCRA, or an approved PCB disposal facility. The generator must provide written notice, including the quantity to be shipped and highest concentration of PCBs, at least 15 days before the first shipment of bulk PCB remediation waste from each cleanup site by the generator, to each off-site facility where the waste is destined for an area not subject to a TSCA PCB Disposal Approval.

Maintain a written record of all sampling and analysis of PCBs or notifications for 5 years from the date of the waste generation.

Risk-based Option

The EPA Regional Administrator approves case-by-case, risk-based cleanup, storage, or disposal of PCB remediation waste as an alternative to other options.

5.6.6 Liquid PCB Waste ≥ 50ppm

PCB liquids at concentrations ≥50 ppm must be disposed of in an EPA-approved incinerator. PCB liquids at concentrations between 50 ppm and 500 ppm may be disposed of in a high efficiency boiler. Liquids from incidental sources, such as precipitation, condensation, leachate, or load separation and that are associated with PCB Articles or non-liquid PCB wastes, may be disposed of in an EPA-approved chemical waste landfill if the liquids are < 500 ppm PCBs and are not ignitable.

5.6.7 PCB Articles

PCB articles with concentrations at 500 ppm or greater must be disposed of in an incinerator, or in a chemical waste landfill, provided that all free-flowing liquid PCBs have been thoroughly drained from any articles before the articles are placed in the chemical waste landfill, and that the drained liquids are disposed of in an incinerator.

Electrical Equipment

The following are PCB concentration assumptions for electrical equipment:

1. Any person may assume that transformers with <3 pounds (1.36 kilograms) of fluid, circuit breakers, reclosers, oil-filled cable, and rectifiers whose PCB concentration is not established contain PCBs at <50 ppm.
2. Any person must assume that mineral oil-filled electrical equipment that was manufactured before July 2, 1979, and whose PCB concentration is not established is PCB-contaminated electrical equipment (i.e., contains >50 ppm PCB, but <500 ppm PCB). All pole-top and pad-mounted distribution transformers manufactured before July 2, 1979, must be assumed to be mineral-oil filled. It may be assumed that electrical equipment manufactured after July 2, 1979, is non-PCB (i.e., <50 ppm PCBs). If the date of manufacture of mineral oil-filled electrical equipment is unknown, the equipment must be assumed to be PCB-contaminated.
3. A transformer manufactured prior to July 2, 1979, that contains 1.36 kg (3 pounds) or more of fluid other than mineral oil and whose PCB concentration is not established, must be assumed to be a PCB transformer (i.e., >500 ppm). If the date of manufacture or the type of dielectric fluid is unknown, it must be assumed that the transformer is a PCB transformer.
4. A capacitor manufactured prior to July 2, 1979, whose PCB concentration is not established must be assumed to contain > 500 ppm PCBs. Any person may assume that a capacitor manufactured after July 2, 1979, is non-PCB (i.e., <50 ppm PCBs). Any person may assume that a capacitor marked at the time of manufacture with the statement ``No PCBs'' in accordance with Sec. 761.40(g) is non-PCB.

Transformers containing ≥500 ppm must be registered with EPA as regulated equipment. Transformers discovered later through sampling to be regulated must register within 30 days of discovery. The registration requirement includes transformers in use and stored for potential future use.

Electrical equipment taken out of service are subject to new disposal requirements. Previously, the empty carcass from drained electrical equipment was not regulated and often recycled. The recycling process resulted in the open burning of residual PCBs (which is prohibited). Therefore, EPA now requires owners to remove as much of the PCB liquid as possible and dispose of the empty carcass in an industrial waste landfill.

The removal of PCBs from electrical equipment is considered "processing for disposal" and does not require a TSCA PCB disposal approval. However, dilution cannot occur during processing. EPA has clarified what constitutes "processing for disposal:"

- Draining liquids from equipment
- Transferring liquids from storage for transport
- Dismantling equipment
- Packaging for transport and disposal
- Combining materials

5.6.8 Radioactive PCB Waste

The disposal of radioactive PCB waste must take into account both its PCB concentration and its radioactive properties. If, taking into account only the properties of the PCBs in the waste, the waste meets the requirements for disposal in a facility permitted, licensed, or registered by a state as a municipal or non-municipal non-hazardous waste landfill, then the person may dispose of the radioactive PCB waste, without regard to the PCB component of the waste, on the basis of its radioactive properties in accordance with all applicable requirements for the radioactive component of the waste.

6.0 Training Requirements

Employees handling or managing PCBs must successfully complete the following training:

- Dangerous Goods Training
- Environmental Awareness Training
- Waste Management Training

7.0 Checklists

The "HSE Self-Assessment Checklist—PCB Waste Management in Attachment 1 is provided as a method for verifying compliance with this SOP. The REM specifies the frequency in which this checklist shall be completed by the SC and provides this information in the project's written safety plan. The REM shall assist the SC in resolving any deficiencies identified during the self-assessment. The REM may also use this checklist when performing EHS audits at CH2M HILL projects, including subcontractor's activities.

8.0 References

The following regulations were referenced to prepare this Enterprise Standard Operating Procedure:

- US Environmental Protection Agency (EPA): 40 CFR 761, Toxic Substances Control Act (TSCA)

- US Environmental Protection Agency (EPA): 40 CFR Parts 260 through 279, Resource Recovery and Conservation Act (RCRA)

- US Department of Transportation (DOT): 49 CFR 171 through 180, Hazardous Materials

9.0 Attachments

Attachment 1: **Self Assessment Checklist – PCB Waste Management**

10.0 Approval

Revision	Date	Prepared By	Approved By:
1.0	10-08-2007	Jim Kelly	

CH2MHILL

Attachment 1: HSE Self-Assessment Checklist

CH2MHILL

HS&E Self-Assessment Checklist –PCB Waste Management

This checklist shall be used by CH2M HILL personnel **only** and shall be completed at the frequency specified in the project's HSP/FSI. This checklist is to be used at locations where CH2M HILL employees are exposed to PCB hazards, or are required to perform oversight of a subcontractor whose personnel are exposed to PCB hazards.

CH2M HILL staff shall not direct the means and methods of subcontractor PCB hazard activities nor direct the details of appropriate corrective actions. The subcontractor must determine how to correct deficiencies and CH2M HILL staff must carefully rely on their expertise. Conditions considered to be imminently dangerous (possibility of serious injury or death) must be corrected immediately or all exposed personnel must be removed from the hazard until corrected.

Project Name: _____ Project No.: _____

Location: _____ PM:

Auditor: _____ Title: _____ Date: _____

This specific checklist has been completed to:

☐ Evaluate CH2M HILL compliance with its Waste Management: PCBs program (SOP HSE-412)

☐ Evaluate a CH2M HILL subcontractor's compliance with its PCB Hazard program

Subcontractors Name: _____

- Check "Yes" if an assessment item is complete/correct
- Check "No" if an item is incomplete/deficient. Deficiencies shall be brought to the immediate attention of the subcontractor. Section 3 must be completed for all items checked "No."
- Check "N/A" if an item is not applicable
- Check "N/O" if an item is applicable but was not observed during the assessment

SECTION 1

	Yes	No	N/A	N/O
STORAGE AND TRANSPORT (5.1)				
1. PCB wastes stored for less than 30 days.	☐	☐	☐	☐
2. PCB waste containing ≥50ppm, stored for 30 days is stored in non-leaking containers.	☐	☐	☐	☐
3. PCB waste containing ≥ 50ppm, stored for 30 days has SPCC plan in place for liquids.	☐	☐	☐	☐
4. Liquid PCB waste containing ≥ 50ppm, stored for 30 days is managed in DOT-approved drums or stationary bulk storage tanks.	☐	☐	☐	☐
5. PCB waste containing < 50ppm, stored for <30 days has the generic ID number, "40 CFR PART 761" is written on manifests, records and reports if the facility does not have an EPA ID number.	☐	☐	☐	☐
6. PCB wastes stored for more than 30 days have an EPA ID number.	☐	☐	☐	☐
7. PCB wastes stored for 30 days or more have adequate roof and walls to keep rainfall from waste.	☐	☐	☐	☐
8. PCB wastes stored for 30 days or more have 6-inch continuous curb around the items with a containment volume equal to greater than twice the largest PCB item.	☐	☐	☐	☐

	Yes	No	N/A	N/O
9. PCB wastes stored for more than 30 days in containment area that is constructed of cement or non-porous material.	☐	☐	☐	☐
10. PCB wastes stored for more than 30 days in containment areas that have no drains, expansion joints or other openings which could allow liquid to escape.	☐	☐	☐	☐
11. PCB wastes stored for more than 30 days in containment area located within a 100-year floodplain.	☐	☐	☐	☐
12. PCB equipment and containers are stored in storage area for no longer than 1 year.	☐	☐	☐	☐
13. Covers and liners are provided for bulk PCB remediation waste or PCB bulk product waste can be stored of up to 180 days.	☐	☐	☐	☐
Storage (5.2)				
14. DOT containers/packaging are used for PCB storage.	☐	☐	☐	☐
Labels (5.3)				
15. Regulated PCB wastes, greater than 50 ppm, are marked and labeled using EPA approved labels.	☐	☐	☐	☐
Manifest (5.4)				
16. PCB-regulated waste transported with a manifest.	☐	☐	☐	☐
17. Uniform hazardous waste manifest (EPA Form 8700-22) states the serial or ID number of the PCB waste, the date of removal from service, and the weight in kilograms of the waste.	☐	☐	☐	☐
Transport (5.5)				
18. PCB wastes must be marked, labeled, and transported in compliance with DOT requirements.	☐	☐	☐	☐
Disposal Procedures (5.6)				
19. Disposal options have been determined based on the PCB item and concentration.	☐	☐	☐	☐
20. PCB concentrations for non-liquids have been determined based on a dry weight basis.	☐	☐	☐	☐
21. PCB concentrations for liquids have been determined based on a wet weight analysis.	☐	☐	☐	☐
22. Consult disposal requirements of PCB waste < 50 ppm.	☐	☐	☐	☐
23. EPA must be notified at least 30 days prior to clean up.	☐	☐	☐	☐
24. Levels have been determined based on the kind of material and potential exposure left after cleanup has been completed.	☐	☐	☐	☐
25. PCB bulk product waste disposal options determined.	☐	☐	☐	☐
26. PCB small capacitors disposal options determined.	☐	☐	☐	☐
27. Remediation waste disposal options determined.	☐	☐	☐	☐
28. Maintain written record of all sampling and analysis of PCBs for 5 years from the date of the waste generation.	☐	☐	☐	☐
29. Consulted EPA Regional Administrator for approval for risk-based cleanup, storage, or disposal of PCB remediation waste.	☐	☐	☐	☐

		Yes	No	N/A	N/O
30.	Liquid PCB waste concentrations and disposal options determined.	☐	☐	☐	☐
31.	PCB articles concentrations and disposal option determined.	☐	☐	☐	☐
32.	Electrical equipment PCB concentrations and disposal option determined.	☐	☐	☐	☐
33.	Radioactive PCB waste concentrations and disposal option determined.	☐	☐	☐	☐

SECTION 3 Complete this section for all items checked "No' in Sections 1 or 2. Deficient items must be corrected in a timely manner.		
Item #	**Corrective Action Planned/Taken**	**Date Corrected**

Auditor: _____ Project Manager: _____

STANDARD OPERATING PROCEDURE

Miniram Personal Monitor

I. Purpose

The purpose of this SOP is to provide general reference information using the Miniram (Mini Real-time Aerosol Monitor) Model PDM-3 to monitor airborne particulates.

II. Scope

This procedure provides information regarding the field operation and general maintenance of the Miniram Model PDM-3. The information contained herein presents the operation procedures for this field monitoring equipment. Review of the owner's instruction manual is a necessity for more detailed descriptions pertaining to the operation and maintenance of the monitor.

III. Definitions

Miniram - Mini Real-time Aerosol Monitor (Miniram) Model PDM-3 used to monitor airborne particulates for preferential response to the particle size range of 0.1 to 10 micrometers.

Sun Shield -.An accessory to the Miniram that protects the sensing elements from excessive ambient light fluctuations.

Z-Bag kit - A calibration kit that consists of a one-way flow rubber bulb for manual air pumping, a filter cartridge, a zippered plastic container, and connecting hardware.

mg/m3 - milligrams per cubic meter of particulate (size range of 0.1 to 10 micrometers) in air, by volume

IV. Responsibilities

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other approved procedures are developed The Project Manager is responsible for selecting qualified individuals for the monitoring activities.

Health and Safety Officer - The Health and Safety Officer is responsible for developing a site-specific Health and Safety Plan (HASP) which specifies air monitoring requirements.

Field Team Leader - It is the responsibility of the Field Team Leader to implement these procedures in the field, and to ensure that the field team performing air monitoring activities, have been briefed and trained to execute these procedures before the start of site operations.

Site Safety Coordinator (SSC) - The SSC is responsible for ensuring that the specified air monitoring equipment is on site, calibrated, and used correctly by the field team. The SSC will coordinate these activities with the Field Team Leader.

Field team - It is the responsibility of the field team to follow these procedures or to follow documented project-specific procedures as directed by the Field Team Leader/SSC. The field team is responsible for documenting the air monitoring results in the field logbook during field investigation.

V. Procedures

The following sections provide information on the operating principles, calibration, operation, and maintenance of the Miniram.

A. Principle of Operation

The Miniram Model PDA13 (Miniram) is an airborne particulate monitor whose operating principle is based on the detection of scattered electromagnetic radiation in the near infrared. The Miniram uses a pulsed light emitting source which generates a narrow-band emission centered at 880 nanometers (=). It is a light scattering aerosol monitor of the nephelometric type (i.e., the instrument continuously senses the combined scattering from the population of particles present within its sensing volume [approximately 1 cm3] whose dimensions are large compared with the average separation between the individual airborne particles.)

In the "Open Sensing Chamber Sampling Method", air surrounding the Miniram passes freely through the open aerosol sensing chamber as a result of air transport caused by convection, circulation, ventilation and personnel movement (i.e., a pump is not used). The scattering sensing parameters have been designed for preferential response to the particle size range of 0.1 to 10 micrometers, ensuring high correlation with standard gravimetric measurements of both the respirable and inhalable size fractions. The rate at which air passes through the sensor does not influence the indicated concentration because the detection is performed directly on every parcel of air traversing the fixed sensing volume. Therefore, flow velocity through a real-time sensor such as the Miniram influences only the response time.

The Miniram measures the concentration of any airborne particles, both solid and liquid, and the display indicates this level in the units of milligrams per cubic meter (mg/m3), based on its factory calibration. The Miniram should be operated in a vertical position and away from reflecting surfaces.

The Miniram comes with a sun shield accessory that protects the sensing elements from excessive ambient light fluctuations. The sun shield is used for outdoor use and under fluctuating bright light illumination. However, it is advisable to use the sun shield at all times.

B. Calibration

Calibration is achieved in the field using a Z-Bag TM Calibrator which will provide a clean-air environment inside a plastic bag into which the Miniram is placed for zeroing. The Z-Bag kit consists of a one-way flow rubber bulb for manual air pumping, a filter cartridge, a zippered plastic container, and connecting hardware.

Prior to calibration, ensure that the Miniram is clean before placing into Z-Bag. Do not expose Z-Bag to sub-zero freezing temperatures as the plastic zippered bag may crack.

The calibration procedure is as follows:

1. Place Z-Bag on flat surface with red flow fitting facing up. Flatten bag. Remove small plastic cap from flow fitting on bag.
2. Connect rubber bulb/filter assembly to red flow fitting of plastic bag, until flush with bottom of fitting.
3. Miniram should be in its OFF condition (check display). If display is blanked, or if Miniram is in another mode, press OFF.
4. Open Z-Bag and place Miniram inside at the center. Press ZERO* and immediately zip closed the bag and begin pumping the rubber bulb/filter assembly.
5. Inflate Z-Bag up to a height of about five inches, then maintain the bag pressure until the Miniram displays OFF again.
6. Record background reading displayed while zeroing, on calibration form.
7. Unzip Z-Bag and remove Miniram. Place rubber bulb/filter assembly inside Z-Bag, and plug small plastic cap into flow fitting to close it. Zip close while flattening Z-Bag to ensure cleanliness of the bag interior.
8. Miniram is ready for use.

The "zero value" is the background level and is automatically subtracted from all aerosol concentrations readings during the measurement mode. Therefore, the displayed readings depend only on the actual dust concentration present within the sensing chamber. It will increase somewhat as the chamber inner walls and windows become contaminated with dust. A **zero** value greater than 3 mg/m2 indicates excessive chamber contamination. For cleaning instructions, refer to manufacturer's operating manual.

C. Use and Applications

To use the Miniram, remove it from the case and observe the display. If the display is blank the Miniram is in the minimum power mode. An "OFF" display means that it has been in the off mode for less than 48 hours.

Depending on the mode of interest, refer to the subsections below for a brief explanation of the use and applications.

1. Measure (MEAS) Mode

With a blank display mode press OFF and wait until the display reads "OFF" (approximately 5 seconds), before pressing MEAS to initiate measurement cycle. - If the Miniram shows "OFF", press MEAS directly to initiate measurement cycle.

The first readout displayed is "GO" (or "CGO" if TIME is also pressed), followed by the last concentration reading or ".00". Approximately 36 seconds after pressing MEAS the first new 10 second averaged concentration reading is displayed. All subsequent readings are concentration values in milligrams per cubic meter, updated every 10 seconds. The Miniram will run in this mode for 500 minutes after which it will stop and display the OFF reading (retaining in storage the concentration average and elapsed time information).

Once the measurement cycle has started, the only way it will be stopped is by pressing OFF. The Miniram normally operates in the .00 to 9.99 mg/m3 range but whenever a 10-second concentration exceeds 9.99 mg/m3 it will automatically switch to a .0 to 99.9 mg/m3 range and remain their until the concentration drops to the lower range.

If both MEAS and TIME are pressed at the same moment (TIME then MEAS) the Miniram will display "CGO" (for Continuous Go) which will cause the instrument to measure continuously in 500 minute intervals. It will run continuously until OFF is engaged or the batteries are exhausted at which time "OFF" will be displayed. Concentration averages and timing information for the last seven 500 minute intervals will remain in storage.

2. Time-Weighted Average (TWA) Mode

During the measurement mode, if TWA is pressed the display will indicate the average concentration in mg/m^3 up to that instant, from the start of the last run. The value of TWA is updated every 10 seconds. After releasing the TWA key the Miniram display returns to the 10-second concentration display.

3. Shift-Average (SA) Mode

During the measurement mode, if SA is pressed the display will provide the aerosol concentration up to that moment, averaged over an 8-hour shift period. This concentration corresponds to the exposure from the start of the measurement cycle and is updated every 10 seconds. After releasing the SA key the Miniram display returns to the 10-second concentration display.

4. Play Back (PBK) Mode

With the Miniram in the off mode, the stored information can be played back by pressing PBK for more than 1 second. The information will be played back in the following order:

- ID number
- Shift or run number
- Sampling time in minutes
- Off-time between the last and next run (in tens of minutes)

- Average in mg/m^3

This sequence is repeated seven times; an average reading of 9.99 mg/m^3 indicates that a significant overload condition occurred during that run. It will take approximately 70 seconds to run through this program.

5. ID Number Selection

In order to change the Miniram identification number, press the OFF key then the ID# key and the presently stored number (between 1 and 999) will be displayed. The raise the number press the up arrow key, and to lower the number press the down arrow key. Pressing the OFF key after this selection will lock-in that number.

D. Maintenance

After each use, the Miniram should be wiped clean with a soft cloth and connected to charger. The Miniram requires a minimum 8 hour charge for daily operation. When not in use it should be stored in the accompanying case to avoid particulate build up in the sensing chamber.

When the zero value exceeds 3 mg/m^3, the sensing chamber may need to be cleaned following the instructions provided in the manufacturer's operating manual.

VI. Quality Assurance Records

Quality assurance records will be maintained for each air monitoring event. The following information shall be recorded in the Field Logbook.

- Identification - Site name, date, location, CTO number, activity monitored (monitoring --well installation, etc.), serial number, time, resulting concentration, comments and identity of air monitoring personnel.
- Field observations - Appearance of sampled media (if definable).
- Additional remarks (e.g., the Miniram experienced a wide range of fluctuations).

VII. References

Monitoring Instruments for the Environment (MIE), INC., Miniram Personal Monitor

Model PDM-3 Operations Manual. March 1990.

QEC
Quality Environmental Containers

P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	_____ GRAB _____ COMPOSITE

ANALYSIS REQUESTED

QEC
Quality Environmental Containers

P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	_____ GRAB _____ COMPOSITE

ANALYSIS REQUESTED

QEC
Quality Environmental Containers

P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	_____ GRAB _____ COMPOSITE

ANALYSIS REQUESTED

QEC
Quality Environmental Containers

P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	_____ GRAB _____ COMPOSITE

ANALYSIS REQUESTED

CUSTODY SEAL

QEC
Quality Environmental Containers
800-255-3950 • 304-255-3900

DATE _____
SIGNATURE _____

CUSTODY SEAL

QEC
Quality Environmental Containers
800-255-3950 • 304-255-3900

DATE _____
SIGNATURE _____

CUSTODY SEAL

QEC
Quality Environmental Containers
800-255-3950 • 304-255-3900

DATE _____
SIGNATURE _____

CUSTODY SEAL

QEC
Quality Environmental Containers
800-255-3950 • 304-255-3900

DATE _____
SIGNATURE _____

STANDARD OPERATING PROCEDURE

Shallow Soil Sampling

I. Purpose

To provide general guidelines for the collection and handling of surface soil samples during field operations.

II. Scope

The method described for surface soil sampling is applicable for loosely packed earth and is used to collect disturbed-soil samples.

III. Equipment and Materials

- Sample jars.
- A hand auger or other device that can be used to remove the soil from the ground. Only stainless steel, Teflon, or glass materials should be used. The only exception is split spoons, which are most commonly available in carbon steel; these are acceptable for use only if they are not rusty.
- A stainless steel spatula should be used to remove material from the sampling device.
- Unpainted wooden stakes or pin flags
- Fiberglass measuring tape (at least 200 feet in length)

IV. Procedures and Guidelines

A. Wear protective gear, as specified in the Health and Safety Plan.

B. To locate samples, identify the correct location using the pin flags or stakes. Proceed to collect a sample from the undisturbed soil adjacent to the marker following steps C and D. If markers are not present, the following procedures will be used.

1. For samples on a grid:

   a. Use measuring tape to locate each sampling point on the first grid line as prescribed in the sampling plan. As each point is located, drive a numbered stake in the ground and record its location on the site map and in the logbook.

   b. Proceed to sample the points on the grid line.

c. Measure to location where next grid line is to start and stake first sample. For subsequent samples on the line take two orthogonal measurements: one to the previous grid line, and one to the previous sample on the same grid line.

d. Proceed to sample the points on the grid line as described in Section C below.

e. Repeat 1c and 1d above until all samples are collected from the area.

2. For non-grid samples:

a. Use steel measuring tape to position sampling point at location described in the sampling plan by taking two measurements from fixed landmarks (e.g., corner of house and fence post).

b. Note measurements, landmarks, and sampling point on a sketch in the field notebook, and on a site location map.

c. Proceed to sample as described in Section C below.

d. Repeat 2a through 2c above until all samples are collected from the area.

C. To the extent possible, differentiate between fill and natural soil. If both are encountered at a boring location, sample both as prescribed in the field sampling plan. Do not locate samples in debris, tree roots, or standing water. In residential areas, do not sample in areas where residents' activities may impact the sample (e.g., barbecue areas, beneath eaves of roofs, driveways, garbage areas). If an obstacle prevents sampling at a measured grid point, move as close as possible, but up to a distance of one half the grid spacing in any direction to locate an appropriate sample. If an appropriate location cannot be found, consult with the Field Team Supervisor (FTS). If the FTS concurs, the sampling point will be deleted from the program. The FTS will contact the CH2M HILL project manager (PM) immediately. The PM and Navy Technical Representative (NTR) will discuss whether the point should be deleted from the program. If it is deleted, the PM will follow-up with the NTR in writing.

D. To collect samples:

1. Use a decontaminated stainless steel scoop/trowel to scrape away surficial organic material (grass, leaves, etc.) adjacent to the stake. New disposable scoops or trowels may also be used to reduce the need for equipment blanks.

2. If sampling:

a. Surface soil: Obtain soil sample by scooping soil using the augering scoop/trowel, starting from the surface and digging down to a depth of about 6 inches, or the depth specified in the workplan.

b. Subsurface soil: Obtain the subsurface soil sample using an auger down to the depths prescribed in the field sampling plan.

3. Take a photoionizaton detector (PID) reading of the sampled soil and record the response in the field notebook. Also record lithologic description and any pertinent observations (such as discoloration) in the logbook.

4. Empty the contents of the scoop/trowel into a decontaminated stainless steel pan.

5. Repeat this procedure until sufficient soil is collected to meet volume requirements.

6. For TCL VOC and field GC aliquots, fill sample jars directly with the trowel/ scoop and cap immediately upon filling. DO NOT HOMOGENIZE.

7. For TCL pesticides/PCBs and SVOCs, TAL metals, and field XRF aliquots, homogenize cuttings in the pan using a decontaminated stainless steel utensil in accordance with SOP *Decontamination of Drilling Rigs and Equipment*.

8. Transfer sample for analysis into appropriate containers with a decontaminated utensil.

9. Backfill the hole with soil removed from the borehole. To the extent possible, replace topsoil and grass and attempt to return appearance of sampling area to its pre-sampled condition. For samples in non-residential, unmowed areas, mark the sample number on the stake and leave stake in place. In mowed areas, remove stake.

V. Attachments

None.

VI. Key Checks and Items

- Use phthalate-free latex or surgical gloves and other personal protective equipment.
- Transfer volatiles first, avoid mixing.
- Decontaminate utensils before reuse, or use dedicated, disposable utensils.

STANDARD OPERATING PROCEDURE

Soil Sampling for VOCs Using the EnCore® Sampler

I. Purpose and Scope

The purpose of this procedure is to provide guidelines for obtaining samples of surface and subsurface soils using the EnCore® Sampler.

II. Equipment and Materials

- The EnCore® Sampler 5g or 25g versions
- Reusable T-handle with a plunger
- 40 mL VOA vial
- 2 oz wide mouth jar

III. Procedures and Guidelines

The sampling point is located and recorded in the field logbook. Debris should be cleared from the sampling location. The EnCore® sampler is being used to collect, store and deliver soil in a sealed, headspace-free state.

A. Surface and Shallow Subsurface Sampling

A shovel, post-hole digger, or other tool can be used to remove soil to a point just above the interval to be sampled. Remove EnCore® sampler from package and attach handle. Quickly collect a 5 or 25 gram sample using the EnCore® sampler. Attach the cap. Fill out a label and attach to sampler.

Ship one EnCore® Sampler per sample location. If low-level analyses are needed, two additional EnCore® samplers will be required. T**he EnCore® sampler has to reach the lab for preservation within 48 hours.** Please refer to the SOP *Packaging and Shipping Procedures* for guidance on shipping.

The soils removed from the borehole should be visually described in the field log book, including approximated depths.

When sampling is completed, photo-ionization device (PID) readings should be taken directly above the hole, and the hole is then backfilled.

B. Split-Spoon Sampling

Using a drilling rig, a hole is advanced to the desired depth. For split-spoon

sampling, the samples are then collected following the ASTM D 1586 standard (see SOPs for *Soil Boring Drilling and Abandonment* or *Logging of Aoil Borings* for this ASTM). The sampler is lowered into the hole and driven to a depth equal to the total length of the sampler; typically this is 24 inches. The sampler is driven in 6-inch increments using a 140-pound weight (``hammer'') dropped from a height of 30 inches. The number of hammer blows for each 6-inch interval is counted and recorded. To obtain enough volume of sample for subsequent laboratory analysis, use of a 3-inch ID sampler may be required. Blow counts obtained with a 3-inch ID spoon would not conform to ASTM D 1586 and would therefore not be used for geotechnical evaluations.

Once retrieved from the hole, the sampler is carefully split open. Care should be taken not to allow material in the sampler to fall out of the open end of the sampler. To collect the sample, the surface of the sample should be removed with an empty EnCore® Sampler. Samples collected for volatiles analysis should be placed directly into the sample containers from the desired depth in the split spoon.

Split-spoon samples also will be collected using a tripod rig. When using a tripod rig the soil samples are collected using an assembly similar to that used by the drilling rig.

IV. Attachments

None

V. Key Checks and Preventative Maintenance

Check that sample collection is swift to avoid loss of volatile organics during sampling.

Appendix C
Data Management Documents

DRAFT

DATA MANAGEMENT PROCESS OVERVIEW FOR THE NAVY CLEAN PROGRAM

Prepared 5 May 2006

Prepared by

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1.0 Introduction

This Data Management Process Overview summarizes CH2M HILL's data management protocol in support of the Navy Clean Program.

The Overview is broadly applicable to the management and dissemination of data generated during environmental investigations. It is intended to be a living document and will be amended or revised to accommodate changes in the scope of environmental investigation or data management requirements.

During field investigations for the Navy Clean Program, CH2M HILL will collect a variety of environmental information that will support data analysis, reporting, and presentation. To ensure quality assurance/quality control (QA/QC) and meet current regulatory requirements, a complete audit trail of the information flow must be established. Each step in the data management process (data collection, storage, and analysis) must be adequately planned, executed, and documented. This Overview will describe in detail the specific processes that will be used by the Data Management team to capture, perform QA/QC reviews, manage/track and report the data associated with the Navy Clean Program.

This DMP is composed of 8 sections. Section 1 of this document introduces the Data Management Process. Section 2 discusses the organization of the CH2M HILL EIMS team. Section 3 discusses the data management role in Project Planning and Setup. Section 4 describes the data management role in Sample Collection and Management. Section 5 discussses the data management activities involved in Lab Analysis. Section 6 describes the data management role in Data Validation. Section 7 discusses the activities involved in Data Management. Section 8 describes Data Evaluation and Reporting procedures. Appendix A presents tables summarizing and assessing current data management materials.

2.0 Data Management Team Organization

The CH2M HILL data management team will work together to properly execute the data management process. The team model presented here is based on a Project Manager supported directly by key technology staff. The functional responsibilities of the team are described below. The responsibilities are identified by titles but not necessarily individual staff positions. The workflow among the members of the data management team is shown in Figure 1.

The Activity Manager (AM) and **the Project Manager (PM)** are responsible for preparing the work plan, schedule, milestones, and coordinating efforts with the client. The AM/PM may or may not have adequate skills to guide the data management driven aspects of their project. While the AM/PM must be willing to accept guidance from the technology leaders, they do not need to possess the technology skills as a background. The PM also responsible for ensuring

data quality and is brought into the team to perform data QA/QC at various times during the data management process.

The Environmental Information Specialist (EIS) assigned to the project team is responsible for the coordination of new or existing data generated by field activities or provided by laboratory analyses. The EIS oversees contracted analytical and data validation services, ensures that analytical data are complete and consistent, enters field data results into the **Field Data Entry Tool(FDETool)**, and assists the Database Specialist in resolving any data ambiguities. The EIS will conduct verification activities following receipt of electronic data and participate in QA/QC activities to resolve inconsistencies as necessary. The EIS acts as a liaison between the Database Specialist, the PM, and the Project Chemist.

Database Specialists load data into the Environmental database. This includes analytical results from laboratory electronic data deliverables and field data results that have been entered by the EIS into the **FDETool**. The Database Specialists work with the EIS, Program Database Coordinator, and Program Data Management Coordinator to ensure that the data are loaded successfully and following established program standards and procedures.

The Field Team Leaders (FTLs) help prepare the work plan and implement the plan in the field. FTLs assign staff members to sampling teams; assign responsibilities to team members; prepare for and coordinate sampling activities; oversee the collection, recording, and documentation of the field data; and ensure that the chain-of-custody form is completed correctly.

The Project Chemist prepares the laboratory and data validation subcontracts, ensures that the electronic data deliverable was provided in accordance with the contract, assists the EIS in communicating with laboratories and data validators as needed, assists the EIS in interpreting analytical results, assists in designating CAS Numbers to new analytes, and maintains the regulatory criteria in the database.

A Program Database Coordinator (DBC) has overall responsibility for the design, operation, and maintenance of the Environmental Database. The DBC is responsible for the implementation, and evaluation of standard operating procedures to ensure integrity of the enterprise-wide database system. The DBC works directly with the Database Specialist to coordinate the different activity data and to enhance the database tools, and structure as required to increase performance and efficiency for the entire program

The Program Data Management Coordinator (DMC) is responsible for the CH2M HILL data management process at all Navy bases. The DMC manages and tracks data management personnel schedules and deliverables for the Navy program; interacts with the EIS on all aspects of data management activities; provides guidance and coordination to the EIS during resolution of data inconsistencies; coordinates completion of data queries for reports; coordinates database modification efforts with the DBC; is responsible for designing, developing, and implementing standard data entry and data retrieval tools; and leads the data management continuous process improvement investigation.

The IS Operations Lead monitors workload across all IS activities (GIS, Web, and Database) for resource and schedule conflicts, and works with IS resources to make recommendations for process change and improvement.

The IS Program Lead serves as the primary point of contact for the Navy regarding IS issues, coordinates resource requirements with regional the IS Staffing Lead, and provides direction and management to the DBC, DMC, and IS Operations Lead.

3.0 Project Planning & Setup

3.1 Attend the Kick-Off Meeting

Review the **Project Instructions**, assign sample nomenclature, go over the EIS level of effort needed and budget with the PM. Complete **the EIS Questions to Ask at Start of Project** Form and **EIS DM Budget Tracking** Form. Enter project information into the **Projects Currently in DM Tracking Table** at the link \\orion\proj\CLEANII\DATAMGMT\EIS\Projects_Currently_in_DM.xls. This tracking table should be updated/verified daily throughout the data management process.

3.2 Aid in Lab and Data Validator Acquisition

As requested, assist with the creation of the Lab Engineers Estimate, Lab Bidsheet, Lab RFP, Lab Statement of Work (SOW), and the Data Validation Engineers Estimate, Data Validation Bidsheet, Data Validation RFP, and Data Validation SOW based on the **BOA Rates Spreadsheet** and **Established Document Templates**. Submit these documents to the site Project Chemist for review and approval before they are submitted to Contracts.

3.3 Aid in Field Preparation

Inform the lab of sampling schedule. Coordinate with the lab how and when samples will be delivered to the lab (pick up, overnight, drop off). Ensure that the lab is aware of the required turn around times. If requested, order bottle ware and create sample labels. If requested, once the bottles have arrived, review the order to ensure the proper amount and type of equipment has arrived.

Tools Involved in Project Planning and Setup
BOA Rates Spreadsheet
EIS Questions to Ask at Start of Project Form
EIS DM Budget Tracking Form
Established Document Templates
Project Instructions
Projects Currently in DM Tracking Table

4.0 Sample Collection & Management

4.1 Communication with Field Staff and Lab

Communicate with field staff daily during the field event. Help resolve issues that arise in the field (bottle ware shortage, equipment failure, etc). Inform the lab of the shipment dates and the number of coolers or samples being sent. Ensure samples were received in good condition (no breakage, within holding time, within designated temperature). Notify field crew and PM if there were problems with shipment.

4.2 Sample and Documentation Tracking

Create a **Sample Tracking Sheet** and update it as samples are collected using Project Instruction Tables, Chains of Custody (COC), and Lab Login Reports. The **Sample Tracking Sheet** should be updated and kept current throughout the data management process. Perform a 100% Quality Check (QC) on COCs received from the field crew. Inform field crew and/or lab if corrections need to be made. Verify that confirmation sheets/login reports from the lab contain correct information. Coordinate efforts with the lab if information needs to be corrected. As needed, create and file a **Corrections-To-File Letter**. Track samples throughout the data management process. Ensure that labs and validators deliver the Sample Delivery Groups (SDG) on time. Inform the PM if SDGs are late, and remind the lab of late penalties (if any are in place).

All documentation acquired during the data management process, including SOWs, Bids, COCs, Field Notes, **Sample Tracking Sheets**, Login Reports, **Corrections-to-File Letters**, FDETool QC tables, **Post Load Reports**, Invoices, and Communication Logs shall be compiled throughout the process and stored in the appropriate Activity's Project Notebook.

4.3 Field Data Entry Tool

The **FDETool** can be completed at any time during the sampling event timeline, and will be turned in with the data load. After the lab has received the samples and submitted login reports, complete the **Data Request/Needs Form** and email it to the Database Specialist and copy the DMC and back-up Database Specialist to request the **FDETool**. Enter data into the **FDETool** using the **Sample Tracking Sheet**, field log books and COCs. Be as specific as possible with the information entered (check with the PM and/or FTLs if information to be entered is unclear). Once all field data has been entered, run the **FDETool** output reports and QC them according to the **FDET Instructions for Data QC Form** (\\orion\proj\CLEANII\DATAMGMT\EIS\EIS_Reference_Documents). Send the reports to another EIS or PM to review for accuracy.

Northing and Easting information should be requested from the PM, if it is missing in the **FDETool**. This data should be entered into the **FDETool. However, if the FDETool is not**

being utilized, the Northing and Easting data can be formatted into a spreadsheet format, which can be sent along with the load. All stations that have coordinates must be loaded into EnDat, even if GIS has received the coordinates. See the **Survey Coordinates Flowchart** at \\orion\proj\CLEANII\DATAMGMT\EIS\EIS_Forms.

4.4 Track EIS Budget

Use the **EIS DM Budget Tracking Form** to track the number of hours spent on each task as they are performed. Inform the PM if the budget may be exceeded.

Tools Involved in Sample Collection & Management
Corrections to File Letter
Data Request/Needs Form
EIS DM Budget Tracking Form
FDET Instructions for Data QC Form
Field Data Entry Tool (FDETool)
Sample Tracking Sheet
Survey Coordinates Flowchart

5.0 Lab Analysis

5.1 QC Lab Data

Verify that the hard copy data and **Electronic Data Deliverables (EDDs)** are complete and acceptable as outlined in the **EIS QC Checklist for Unvalidated and Validated EDDs and Hard Copy Data Form**. Run a quality check on the EDD columns to ensure basic quality. Perform a 10% check of the analysis results. Ensure that the hard copy data matches the EDD. If errors are found, inform the PM and request corrected data from the lab.

5.2 Communicate with the Lab

Should the EDD be missing data, contact the PM and coordinate efforts with the lab to receive the missing data.

5.3 Run Tables

Communicate with the PM to determine if preliminary raw and detects tables are needed. Should tables be desired, verify the requirements and formatting (i.e. headers, footers, or other

special needs) to be included on the table. Run the **Raw & Detects Tables from Unvalidated or Validated EDD Macro** on data in the EDD to create tables to assist the PM with a preliminary data analysis. A separate table must be created for EACH matrix (solid/aqueous) and sample purpose (Normal, Blanks). Ask the PM how the tables should be run before beginning.

5.4 Hard Copy Management

If data are to be validated, follow the instructions for Hard Copy Management in the Data Validation section, below. If data are not to be validated, hold on to the hard copies until project closeout/completion. After all corrections identified through the data management process have been completed (if any), the final report written, and the project determined complete, gain approval from the PM to archive the hard copy. Note, skip to section 7.0, Data Management, for EDDs that are not to be validated.

5.5 Hard Copy Archiving

If data will not be validated, fill out the **Data Archiving (List of Contents) Form,** located at the link \\Orion\PROJ\CLEANII\DATAMGMT\EIS\Data_Archiving, for each SDG, and attach it to the data packages. Once the PM has granted approval for hard copy archiving at project completion, give the boxes of data to the Data Archiving Specialist. The data will be prepped for archiving and filed within the building until the Data Archiving Specialist has received authorization to send the data to storage.

Tools Involved in Lab Analysis
Data Archiving (List of Contents) Form
EDD
EIS QC Checklist for Unvalidated and Validated EDDs and Hard Copy Data Form
Raw & Detects Tables from Unvalidated or Validated EDD Macro

6.0 Data Validation

6.1 Hard Copy Management

If data are to be validated, the hard copy data, **EDDs**, and a **QC Association Table** will need to be mailed or emailed to the data validator. Photo copy the Form I Summary Package (which should be provided by the lab) before mailing the hard copy, to keep on file while the complete packages is with the validator. Fill out the **Data Archiving (List of Contents) Form** for each SDG, and attach it to the data packages. The **QC Association Table** is created using the COCs, field notes, and the field crew to ensure accuracy. Further instructions on the QC table are located in the form "**QC Association Table**", under

\\orion\proj\CLEANII\DATAMGMT\EIS\EIS_Forms. The **QC Association Table** can be emailed to the data validator along with the EDD. If sending more than one EDD, prepare the EDDs to the validator's preference (i.e. one large file or divided by SDG).

6.2 Communicate with Validator

Let the data validator know ahead of time when to expect data. Inform the validator of any samples or analyses that should not be validated. (i.e. grain size should not be validated). Work with the data validator to coordinate the return of the data package to CH2M HILL for archiving. Once the data package has been returned to CH2M HILL, follow the Hard Copy Archiving procedure above.

6.3 Post-Validation

Review and QC the validated data according to the **EIS QC Checklist for Unvalidated and Validated EDDs and Hard Copy Data Form**. Verify that the validated hard copy data and EDDs are complete and acceptable. Data validators should have added qualifiers to the DV_QUAL and DV_QUAL_CODE fields only. Check the values in the DV_QUAL field against the valid value choices. Perform a 100% check of the DV_QUAL and DV_QUAL_CODE fields. Ensure that the hard copy values match the EDD. Ensure that every record requiring a data validation qualifier has one (i.e. if the Lab_Qual field has a U qualifier then there MUST be a qualifier in the DV_QUAL field).

Run raw and detects tables of the combined EDD using the **Raw & Detects Tables from Unvalidated or Validated EDD Macro**. Check to make sure there are no duplicate results for any of the samples. Send the raw and detects tables, validation report, and validated EDD to the Project Chemist for a "Pre-Load Check."

Comment [CH1]: Anita/Ann were budgeted 4 hours by Phil to complete a checklist for this, and it has not been done yet.

Tools Involved in Data Validation
Data Archiving (List of Contents) Form
EDD
EIS QC Checklist for Unvalidated and Validated EDDs and Hard Copy Data Form
QC Association Table

7.0 Data Management

7.1 Load Preparation

Compile the validated SDG **EDDs** into one Excel file, if they are not formatted as such already. Add in and populate the additional columns CTO, Lab, and Validated at the end of the EDD. Add in a column before Prep_Method called Preparation. Copy and paste the data from Analysis_Method into the Preparation column. Rename the Prep_Method to CH2M_Code, and populate with appropriate valid values. Save the Excel file as an 'Archive EDD' under a new name with the project or event and the date sampling (i.e. "3_CP_CTO-244_GW&SO_103103_ ARCHIVE.xls"). Be as specific as possible when saving the file, as it will become the Archive EDD file.

Create a duplicate copy of the Archive EDD file and save it as the Load EDD (i.e. "3_CP_CTO-244_GW&SO_103103_ LOAD.xls"). In the Load EDD, delete out the surrogate records by deleting ALL records that have a value in the "Result_Type" column. Delete Lab QC Records by deleting ALL records that have a value in the "Lab_QC_Type" column. Remember to save the Load EDD once the modifications are complete.

After the data has been loaded, incorporate any corrections made to the Load EDD by the Database Specialist into the Archive EDD. Mail a copy of the Archive EDD to the DMC to be stored in the archive file (\\orion\proj\CLEANII\DATAMGMT\EDD_Archive).

7.2 Run a Pivot Table

As needed, follow the **Analyte Pivot Table Instructions** file to determine if any analytes are classified under more than one analysis group in the Load EDD. (This step is considered a backup check, as a 'Preferred Analysis Group Check' was performed on the unvalidated EDD, as specified on the **EIS QC Checklist for Unvalidated and Validated EDD and Hard Copy Data Form**.) Use the **Preferred Analysis Group Form** as a reference to assign UNREJECTED results to the correct analysis group for these analytes. If an analyte is not on this list then ask a chemist for assistance and update the **Preferred Analysis Group Form** accordingly.

7.3 PM Review of Data Load

Provide the PM with the cross-tabulated raw and detects tables created from the validated data above, and the Load EDD file. Also ask the PM if they would like a copy of the **Sample Tracking Sheet** or **Project Instructions** to assist with the review.

7.4 Email Data Load

Send the QC'd Load EDD file (the version WITHOUT the surrogate and QC data) and **FDETool** in an email to the Database Specialist for loading into EnDat, and copy the DMC and back-up Database Specialist. In the email, attach an electronic copy of the completed **Data Request/Needs Form** with the following information completed:

- Program Name (ex: Clean II)
- Activity (ex: Little Creek)
- Contract Task Order (CTO)
- Prime Contractor (company responsible for providing a product to the Navy)

- Field Contractor (company who performed the field work)
- Was the data upload scheduled with the DB staff?
- Is the data validated?
- Data Validator Name (If no DV then who within CH2M HILL evaluated the data?)
- Number of samples
- Dates of the sampling event
- Number of records in EDD
- Requested Due Date
- Any Reports Requested?

The Database specialist will then conduct any additional formatting modifications to the EDD as needed to load the data into EnDat.

7.5 Post Load

The Database Specialist shall generate **Post Load Reports** and provide them to the EIS for review and QC. Once the **Post Load Reports** have been QC'd by the EIS, the EIS will then send the reports to the PM for review. Inform the PM of any corrections that need to be made, and coordinate these changes with the Database Specialist. Any changes made to the data by the Database Specialist prior to load, or that will be completed after the load should be tracked, and incorporated into the hard copy and EDD files that are to be archived after project completion.

Tools Involved in Data Management
Data Request/Needs Form
EDD
Field Data Entry Tool (FDETool)
Pivot Table Instructions
Preferred Analysis Group Form
Project Instructions
Raw & Detects Tables from Unvalidated or Validated EDD Macro
Sample Tracking Sheet
Post Load Reports

8.0 Data Evaluation & Reporting

8.1 Run Tables

Meet with the PM to verify table requirements and formatting (i.e. headers, footers, or other special needs). Raw and detects tables must be created for EACH matrix (solid/aqueous). Pull the data from **EnStat**. There are three macro templates that can be utilized to assist with the

formatting of EnStat output files. These include the **Raw, Detects, & Exceedance Tables from EnStat Output Macro**, **HHRA Tables from EnStat Output Macro**, and **EcoRisk Tables from EnStat Output Macro**.

Run the **Raw, Detects & Exceedance Tables from EnStat Macro**, and send the completed tables to the Project Chemist for a final quality check. Provide the completed, QC'd tables to the PM. Other tables can be generated from the remaining macros as requested.

8.2 Review Laboratory and Validator Invoices

Laboratory invoices should be submitted once the laboratory has completed requested analyses, and submitted all results and requested corrections. Data validation invoices should be submitted shortly after the validation has been completed, and the report submitted to CH2M HILL. Invoices will be submitted to the PM through AP Workflow for approval. The PM should then consult the EIS for invoice review before submitting approval. The EIS should review the invoices, and noting any late charges, etc, and update the **Sample Tracking Sheet** accordingly.

8.3 Complete EIS DM Budget Tracking Form

Meet with the PM and the DMC to review the **EIS DM Budget Tracking Form** and discuss lessons learned.

Tools Involved in Data Evaluation & Reporting
EcoRisk Tables from EnStat Output Macro
EIS DM Budget Tracking Form
EnStat
HHRA Tables from EnStat Output Macro
Raw, Detects, & Exceedance Tables from EnStat Output Macro
Sample Tracking Sheet

Appendix A

Summary & Assessment of Data Management Materials

Summary Of Tools Involved In The Data Management Process

Tools	Assessment
BOA Rates Spreadsheet	This is only updated every 5 years. We need an SOP to remind EISs to add a 10% increase for each year after the update year until it is updated again.
Corrections to File Letter	
Data Archiving (List of Contents) Form	Kevin McGarvey, the Archiving Expert will be working in the WDC office through June, and will be stopping by here. He could be tasked to write up an SOP. We might have some mini-SOPs to work from too.
Data Request/Needs Form	Good
EcoRisk Tables from EnStat Output Macro	Good
EDD	Good, though primary keys need revision.
EIS QC Checklist for Unvalidated and Validated EDDs and Hard Copy Data Form	This is a good procedure checklist, and could easily be made into a formal SOP.
EIS Questions to Ask at Start of Project Form	This could use a few formatting tweaks, but is generally good as is.
EIS DM Budget Tracking Form	This should be updated to incorporate all the aspects of the data management process for more accurate tracking
EnDat Post Load Reports	Good. Used to assess and QC data loaded into EnDat to ensure data load accuracy and completeness
EnStat	This needs work to get it running better/correctly. There is a ppt presentation on using this that could serve as a SOP.
Established Document Templates	Currently we work off of pre-existing docs, which vary. Templates must be established.
FDET Instructions for Data QC Form	Needs evaluation
Field Data Entry Tool (FDETool)	Could use a bulk upload function, and built in QC checks

Tools	Assessment
HHRA Tables from EnStat Output Macro	Needs evaluation
Pivot Table Instructions	Could easily be made into a good SOP
Preferred Analysis Group Form	Good
Project Instructions	From PM
Projects Currently in DM Tracking Table	Good
QC Association Table	The example on the server is intended to use as a template, and could use a little tweaking
Raw & Detects Tables from Unvalidated or Validated EDD Macro	This macro could use formatting updates. There is no SOP for this, but I do have a rough mini-SOP that Felicia wrote up.
Raw, Detects, & Exceedance Tables from EnStat Output Macro	Needs evaluation
Sample Tracking Sheet	Need to develop template
Survey Coordinates Flowchart	Good

Summary of Documentation in the Reference Manuals

Document	Assessment for Current DMP	Assessment for NIRIS
IS Personnel 11-2006	Current	Good
Load Process Step by Step	Generic overview, not SOP. Need Bhavana to write a formal SOP if desired	Need New Document
Navy Clean IS Organization	Out of Date	Need New Document
Reference Manual Binder Covers	Current	Good
Ref Manual Page Dividers	Current	Good
Project Manager Role in IS-DM Process	Current	Good
Environmental Information Specialist Role 1	Current	Good
Data Management Coordinator Role	Current	Good
Navy Clean Data Management Process Flowchart	Current	Good
Survey Coordinates Flowchart	Good	Needs Revision
Life of a Sample Flowchart	Needs Revision	Needs Revision
Chemicals in EnDat 010306	Needs periodic updates	Need New Document
Chemical Synonyms in EnDat	Needs periodic evaluation	Need New Document
Common Chemical Synonyms & Abbreviations	Good	Good
Analyses and Methods Commonly Used	Needs periodic updates	Needs periodic updates
FDET Valid Values	Good	Need New Document
Lab Valid Values	Good	Need New Document
DV Valid Values	Good	Need New Document
Field Sample Naming Scheme	Needs Revision (to Sample Nomenclature Protocol for all Bases)	Uncertain
Field Station Naming Scheme	Needs Revision (to Station Nomenclature Protocol for all Bases)	Uncertain
EDD Format CH2M Navy 120605	Needs Updates	Need New Document

Document	Assessment for Current DMP	Assessment for NIRIS
DCLT Manual	None – This is no longer used, as the Tool is broken	Delete
STS Example	Need to develop template	Need to develop template
Corrections To File	Good	Uncertain
Corrections to File Example	Good	Uncertain
FDET Instructions	Good	Delete
FDET Screen Shot	Good	Delete
FDET Stations Report Example	File does not exist	Delete
FDET Sample Report Example	File does not exist	Delete
FDET Field Results Report Example	File does not exist	Delete
FDET Full Detail Report Example	File does not exist	Delete
FDET Result Report in XL Example	Good	Delete
FDET Instructions for Data QC	Needs Evaluation	Delete
Data Management Checklist _rev0306	Needs Revision	Needs Total Revison/Rewrite
Analyte Pivot Table Instructions	Good	Uncertain
Analyte Pivot Table Example	Can not locate file	Uncertain
Preferred Analysis Group	Needs evaluation – have older version (ABL) too	Uncertain
Ex of Pre-Load QC Raw & Detects Tables	Good	Need new document
Ex of Post-Load Station Check Confirmation Rpt from DB Specialist	Cannot locate file	Uncertain
Ex of Post-Load Sample Check Confirmation Rpt from DB Specialist	Cannot locate file	Uncertain
Ex of Post-Load Field Result Check Confirmation Rpt from DB Specialist	Cannot locate file	Uncertain
Ex of Post-Load Analysis Check Confirmation Rpt from DB Specialist	Cannot locate file	Uncertain
EnStat Tool Instructions	PPT, not SOP. Could easily be made into SOP	Need New Tool
EnDat Threshold Criteria	Needs Evaluation	Need New Document
Definitions of RBC & MCL Threshold Variations	Unable to locate Email Doc	Uncertain

Document	Assessment for Current DMP	Assessment for NIRIS
Ex of Unformatted EnStat Post-Load Tables	Good	Need New Document
Ex of Formatted EnStat Post-Load Tables	Good	Need New Document
IS Costing Template 2006Rates 042506	Needs to be Updates	Needs Updating
IS Data Request-Needs Form	Good	Needs Update/New Document
Quarterly Sampling Projection Forms Example	Good	Good
EIS Project Startup Questions_rev0905	Good	Needs Revision
EIS DM Budget Tracking Form	This should be updated to incorporate all the aspects of the data management process for more accurate tracking	Needs Revision
EIS QC Checklist for Unval & Val EDD & Hard Copy Data	Unable to locate document	Needs Revision
EIS Training Checklist	Good	Needs Revision

Data Management Checklist

Base Name:_____ **CTO Number(s):**_____

Site:_____ **PM:**_____

Sample Date Range:_____ **EIS:**_____

Lab:_____ **DV:**_____

SDG #'s:_____

☐ **Attend Kick-off Meeting** (Review project instructions, assign sample nomenclature, go over the EIS level of effort needed and budget with PM). Determine EDD format to be used.

☐ **Aid in field preparation:** Inform lab of sampling schedule, coordinate with the lab how samples will be delivered to the lab (pick up, overnight, drop off) and how often, ensure lab is aware of the required turn around times. If requested, order bottle ware and create sample labels. If requested, once the bottles have arrived, review the order to ensure the proper amount and type of equipment has arrived.

☐ **Communication with field staff and lab during field event:** Communicate with field staff daily. Help resolve issues that arise in the field (bottle ware shortage, equipment failure, etc). Inform the lab of the shipment dates and the number of coolers or samples being sent. Ensure samples were received in good condition (no breakage, within holding time, within designated temperature). Notify field crew and project manager if there were problems with shipment.

☐ **Sample Tracking:**

- Create sample tracking sheet and update it as samples are collected.
- Receive COCs from field crew: Perform a 100% quality check of the chains of custody (COC). Inform field crew and/or lab if corrections need to be made.
- Receive confirmation sheets from lab. Verify they have the correct information. Coordinate with lab if their information needs to be corrected.
- Generate a Corrections-to-File letter for any COC/field log book/login discrepancy. Provide a copy to the laboratory, data validator, Project Manager, and Project Notebook. (See files Instructions_Corrections-to-File, and Template_Corrections-to-File)
- Track samples throughout the data management process. Ensure that labs and validators deliver the Sample Delivery Groups (SDG) on time. Inform project manager if SDGs are late and remind lab of late penalty (if there is one in place).

☐ **Track EIS Budget:** Use the EIS Budget Tracking Form to track the number of hours spent on each task as they are performed. Inform the project manager if you suspect you will go over budget.

Pre-Validation:

☐ **QC lab data (For Specifics see EIS QC Checklist):**

- Verify that the hard copy data and EDDs are complete and acceptable. Run a quality check on the electronic data deliverable (EDD) columns to ensure basic quality.
- Perform a 10% check of the analysis results. Ensure that the hard copy data matches the EDD. If errors are found, inform the project manager and ask the lab to send corrected data.

☐ **Communication with Lab:** If there is missing data from the EDD, contact the project manager and coordinate with the lab to receive the missing data.

☐ **Run Unvalidated Tables:**

- Check with the PM to see if they would like preliminary raw and detects tables. If so, verify the needs on the table. (i.e. headers, footers, or other special needs).
- Run either the "*Raw & Detects Tables from Unvalidated or Validated EDD.xls*" or "*Raw & Detects Tables from Unvalidated or Validated SNEDD.xls*" (depending on EDD format used) macro to create tables to assist the PM in their preliminary analysis of the data.

- Create a separate table for EACH matrix and Site (solid/aqueous).– ask the PM how they would like these tables run before you start.

☐ **Hard Copy Management:** Fill out the Data Archiving Form for the lab data located at the following link and file on the top of each SDG. (\\ariadne\Proj\CLEANII\DataMgmt\EIS\Ref Manual Updating\Data_Mgmt_Manual-Spring-2007)

- The QC association table should be created using the COCs, field notes, and field crew to ensure accuracy. For further instructions on the QC table please go to the link above and select "Template_QC-Association_Table".
- If data are being validated, mail/email the hard copy data, EDDs, and QC association table to the validator. A Form I summary report should also be provided.
- If data are NOT being validated, provide the hardcopy data, EDDs, QC association table, and Unvalidated Raw and Detects tables to the Chemist for an Internal or PreLoad Check (see Chemist Preload Check section below).

☐ **Communication with Validator:** Let the validator know ahead of time when to expect data. Inform validator of any samples or analyses that should not be validated. (For example grain size should not be validated)

Field Data Entry Tool:

☐ **Data Request/Needs Form:** Following sample collection, complete the Data Request/Needs Form and email it to Bhavana Reddy/WDC and copy Mike Zamboni/WDC to request the Field Data Entry Tool (FDETool).

☐ **FDETool Data Entry:** Enter data into the FDETool using field log books and COCs. Be as specific as possible with the information entered (check with the project manager and/or Field Team Leaders if you are unsure of information.)

☐ Request Northing and Easting information from project manager if not provided. This data must be entered into the FDETool. (See file DMP_Survey-Coordinates-Workflow)

☐ **QC FDET Data:** Complete FDET output reports and QC. Send to another EIS or **project manager** to review accuracy.

☐ **NIRIS Field NEDD Output:** Output the necessary NIRIS field-related NEDDs from the FDETool if data is to be loaded into NIRIS (all projects with a SNEDD). Reserve these for after the data has been loaded into EnDat and the data is ready for archiving.

Post-Validation:

☐ **QC validated data (For Specifics see EIS QC Checklist):**

- Verify that the validated hard copy data and EDDs are complete and acceptable
- In the CH2M HILL EDD format, Data validators should have added qualifiers to the DV_QUAL and DV_QUAL_CODE fields and no other fields. In the SNEDD format, data validators should have updated the fields: Analyte_Value, Validator_Qualifier, QC_Narrative, Validator_Name, and Val_Date.
- Check all validation-related Valid Values against Valid Value Look up Tables.
- Perform a 100% check of the DV_QUAL and DV_QUAL_CODE field (or Validator_Qualifier, QC_Narrative fields in the SNEDD). Ensure that the hard copy values match the EDD
- Ensure that every record that needs a data validator qualifier has one (i.e. if the Lab_Qual field has a U qualifier then there MUST be a qualifier in the DV_QUAL or QC_Narrative field)

☐ **Run Validated Tables:**

- Run validated raw and detects tables. Verify any specific needs on the table with the PM. (i.e. headers, footers, or other special needs).

- Run either the "*Raw & Detects Tables from Unvalidated or Validated EDD.xls*" or "*Raw & Detects Tables from Unvalidated or Validated SNEDD.xls*" (depending on EDD format used) macro to create tables to assist the PM and Chemist in their analysis of the data.
- Review tables to determine which analytes are classified under more than one analysis group. Resolve discrepancies with Project Chemist. Or, run a Pivot Table to determine if analytes are assigned to more than one analysis group. Follow the "*Instructions_Analyte-Pivot-.doc*" file to determine which analytes are classified under more than one analysis group. Use the "*Form_Preferred-Analysis-Group.xls*" file as a reference to assign the **UNREJECTED** result to the correct analysis group for these analytes.
- **PM Review of Data:** Provide the tables to the PM. Also ask the PM if they would like a copy of the Sample Tracking Sheet or Project Instructions to assist them in their review.

Archive/PreLoad EDD Prep:

☐ CH2M HILL Format EDDs

- Compile the validated SDG EDDs into one Excel Archive file. In the CH2M HILL EDD format, add in additional columns CTO#, Prep_Method, and Validated. (This file will be archived) Save the Excel load file under a new name with the project or event and the date sampling began (i.e. "PAX_244_GW_Sampling_ARCHIVE_103103.xls"). Be as specific as possible when saving the file.
- For Sample_IDs with DL, RA, or RE appended (this indicates a second run on the same sample and therefore two or more sets of results), the data validator should have selected one result to be used and placed an "R" for rejected on the result(s) not to be used. Check that there is only one usable result for each analyte.
- Remove the DL-, RA-, or RE-type suffixes from all Sample_IDs. Verify that these designations are captured in the "ReRun" column before deletion.
- Conduct one quick final check for anomalies by setting filters for each column.

☐ SNEDD Format EDDs

- Compile the validated SDG EDDs into one Excel file. Save the Excel file under a new name, with the project or event and the date sampling began (i.e. "PAX_244_GW_Sampling_Validated_PreLoad_SNEDD.xls")

Chemist PreLoad Check:

☐ **Project Chemist QC of data**

- If data are NOT being validated, provide the hardcopy data, Archive or PreLoad EDDs, QC association table, and Unvalidated Raw and Detects tables to the Chemist for an Internal or PreLoad Check. Verify that the hard copy data and EDDs are complete and acceptable.
- If data have been validated, provide the hardcopy data validation report, validated EDDs, QC association table, and Validated Raw and Detects tables to the Chemist for an Internal or PreLoad Check. Verify that the validated hard copy data and EDDs are complete and acceptable.

Load Preparation:

☐ CH2M HILL Format EDDs

- Conduct one quick final check of the Updated/QC'd ARCHIVE EDD provided by the Chemist for anomalies by setting filters for each column.
- **Delete Surrogate Records.** Delete ALL records that have a value in the "Result_Type" column.
- **Delete Lab QC Records.** Delete ALL records that have a value in the "Lab_QC_Type" column.
- Save the EDD with the deleted records as the Load EDD. i.e. "PAX_244_GW_Sampling_LOAD_103103.xls")

☐ SNEDD Format EDDs

- Conduct one quick final check of the Updated/QC'd PreLoad EDD provided by the Chemist for anomalies by setting filters for each column. Import the file into the Archive and Load Prep Tool (ALPTool).
- Run the following functions: Prepare ALPTool, Import SNEDD, Check Analyte IDs, Archive EDD, Load EDD, NEDD. Save the outputted EnDat Archive, EnDat Load, and NIRIS NEDD files.

☐ **Email Data Load:** Send the CH2M HILL Excel LOAD file (the version WITHOUT the surrogate and QC data) and FDETool in an email to Bhavana Reddy and copy Mike Zamboni/WDC for loading into EnDat. In the email attach an electronic copy of the completed Data Request/Needs Form with the following information completed:

- Program Name (ex: Clean II)
- Activity (ex: Little Creek)
- CTO
- Prime Contractor (company responsible for providing a product to the Navy)
- Field Contractor (company who performed the field work)
- Was the data upload scheduled with the DB staff?
- Is the data validated?
- Data Validator Name (If no DV then who within CH2M HILL evaluated the data?)
- Number of samples
- Dates of the sampling event
- Number of records in EDD
- Requested Due Date
- Any Reports Requested?

Post Load:

☐ **QC Post Load Reports:** Receive the post load reports from Bhavana Reddy or Mike Zamboni. QC reports and also send the reports to the project managers to review. Inform project managers of any corrections that need to be made. Coordinate these changes with Bhavana or Mike.

☐ **EDD Archiving:** Incorporate any corrections made by Bhavana or Mike during loading, or resolved after loading into the CH2M HILL ARCHIVE EDD, SNEDD, and NIRIS NEDD. Send both files, along with the FDETool NIRIS field-related NEDD files to Chelsea Bennet/VBO for archiving.

☐ **Run Tables:**

- Meet with the PM to verify the needs on the tables (i.e. headers, footers, or other special needs).
- A raw and detects table must be created for EACH matrix and site (solid/aqueous). Ask the PM how they would like these tables run before you start.
- Pull the data from EnStat
- Run the "*Raw, Detects & Exceedance Tables from EnStat.xls*"

☐ **Review Laboratory and Validator Invoices:** Track invoices and approval dates on the tracking sheet

☐ **Complete EIS Budget Form:** Meet with project manager and the data management coordinator to discuss lessons learned.

☐ **File Project Paperwork:** File all documentation generated during the project (STS, Corrections to file, COCs, Lab Logins, etc) into the appropriate project binder for future reference.

EDD Prep for Load and Archive Files

CH2M HILL EDDs

1. Combine EDDs into one EDD. Two versions of the EDD will be generated (I. Archive, and II. Load)
2. ADD 3 Columns to the end of the EDD:
    a. CTO (List the numerical value, or full TO number. Ex. CTO-177 = 0177, TO-45 = TO-45)
    b. Lab (Write out the complete name)
    c. Validated (T or F)
3. Rename the field Preparation as "CH2M_Code". Filter on each Analysis Group, and update this field according to the correct Valid Values for CH2M_Code. Ex. NONS for SVOA, NONE for METAL.
4. Highlight and Copy the Analysis_Method field, and insert the column before the field CH2M_Code. Rename the new field as Preparation.
5. Generate Archive EDD - Save the file as the Archive EDD, with a descriptive file name explaining the data contained therein. Ex file name = CP_CTO-74_Site-30_SD_May-07_ARCHIVE_EDD
6. Generate Load EDD
    a. Remove Lab QC data from the EDD – Filter for non-blanks on the field Result_Type, and delete listed records. Filter for non-blanks on the field Lab_QC_Type, and delete listed records.
    b. Review the field Sample_ID and ensure that all Lab QC Samples have been removed.
    c. Save the file as the Load EDD. Ex. File name = CP_CTO-74_Site-30_SD_May-07_LOAD_EDD

SNEDDs

7. Combine SNEDDs into one SNEDD. Three file versions will be generated (I. Archive EDD, II. Load EDD, and III. NIRIS NEDD)
8. Open the ALPTool, and follow the directions and buttons listed in steps 1 through 4
    a. Step 1 – a) Click on the button 'Prepare ALPTool'. b) Click on the folder button, selected the SNEDD file to be imported from the appropriate director, and click on the button 'Import SNEDD'
        i. If File will not import, clean the SNEDD file: Save the SNEDD as a tab delimited text file. Open a new excel workbook. Go to Data/Import External Data/Import data, and follow the wizard to import the text file into excel. When selecting the fields to import, be sure to specify the formatting type for each as Text. Save the new file, and return to step 1a.
    b. Step 2 – Click on the button 'Check Analyte IDs'. If any records are listed in the pop up, resolve any incorrect analyte IDs.
    c. Step 3 – a) Click on the button 'Archive EDD'. Follow the directions in the pop up to save the file. B) Click on the button 'Load EDD'. Follow the directions in the pop up to save the file.
    d. Step 4 – Click the button 'NEDD'. Follow the directions in the pop up to save the file. Click the button 'Close' to close the entire application.
9. Save Files
    a. Save the Archive EDD. Ex file name = CP_CTO-74_Site-30_SD_May-07_ARCHIVE_EDD
    b. Save the Load EDD. Ex file name = CP_CTO-74_Site-30_SD_May-07_LOAD_EDD
    c. Save the NIRIS EDD. Ex file name = CP_CTO-74_Site-30_SD_May-07_NIRIS_NEDD

EIS QC Checklist for Unvalidated and Validated EDDs/SNEDDs and Hard Copy Data

Sample Tracking Sheet/Lab Login Reports

☐ Compare COCs and STS to the Project Instruction tables to verify that the COCs and Lab Logins reflect all requested analyses. Check with PM/Lab if there are any discrepancies.

Unvalidated EDDs and Hard Copy Data

☐ Compare the analyses reported in the EDD to the STS to very that the lab has reported all requested analyses for each sample. Resolve any discrepancies with the PM/Lab/Chemist.

☐ Verify columns on the SNEDD/EDD match the CH2M HILL format specified; (Header spellings, order)

☐ Check EDD/SNEDD Columns for correct Valid Values

- ☐ Sample IDs match the STS exactly
- ☐ Analysis Groups are correct (ex. Perchlorate can be WCHEM or EXPLO)
- ☐ Dates/Times are numerical and progress appropriately (ex. Date analyzed after date sampled)
- ☐ Chem Names and Analyte ID (CAS #)
- ☐ Ana Values (SNEDD Original Analyte Value) match appropriate Reporting Limits (Metals match MDL, all else match DL)
- ☐ Analyte Values are appended to each Parameter. (Filter for blanks)
- ☐ Units are correct for media (solid vs. aqueous)
- ☐ Result Type, Lab QC Type, Rerun and Lab Code
- ☐ Lab Code and/or Lab Name

Additional SNEDD Valid Values

- ☐ Contract ID, DO CTO Number, and Installation ID are correct
- ☐ CH2M_Code
- ☐ Sample Basis
- ☐ Sample Medium
- ☐ GC Column Type
- ☐ Analysis Result Type
- ☐ QC Control Limit Code

☐ EDD vs. Hard Copy Check. Perform a 10% - 20% QC on each analysis group to verify that the EDD and hard copy are an exact match. Verify results for reextraction/reanalyses are reported in Hardcopy and EDD. Resolve any discrepancies with the Lab/Chemist.

Validated EDDs and Hard Copy Data

☐ Validated EDD columns DV_Qual & DV_Qual_Code contain correct values

☐ For SNEDD format, **Analyte_Values are correctly copied/updated from Original_Analyte_Value. Columns Validator_Qualifier, Validator_Name, and QC_Narrative are populated correctly**

☐ EDD vs. validated marked up Form Is. Perform a 100% QC of all validated data in EDD and hard copy Form Is. Compile a list of discrepancies and request clarification from validator. Review data validation case narratives to help clarify possible mistakes.

☐ Filter on non-detects (U, UJ, UL) in the Lab_Qualifier column and **verify that the validator has copied over all non-detects into the DV_Qual (or Validator Qualifier) column**.

☐ Generate validated Raw and Detects tables from the Validated EDD. Provide validated tables and EDD to Chemist for PreLoad Check.

EDD Prep for Raw and Detects Tables for Unvalidated or Validated Data

1. Combine EDDs into one EDD, and save file as "Combined Unval EDD for Tbl Macro". (Note – SNEDDs and EDDs are different formats and cannot be combined together.
2. Remove Lab QC data from the EDD
    a. CH2M HILL EDD – Filter for non-blanks on the field Result_Type, and delete non-blank records. Filter for non-blanks on the field Lab_QC_Type, and delete non-blank records.
    b. SNEDD – Filter for records that do not equal TRG in the field Analysis_Result_type, and delete records. Filter for records that do not equal REG in the field Lab_QC_Type, and delete records.
3. Select and Open the correct table formatting macro
    a. CH2M HILL EDD – Open "Raw & Detects Tables From Unvalidated or Validated EDD - new EDD format- 04-12-02.xls". When prompted, select Enable Macros.
    b. SNEDD - Open "Raw & Detects Tables From Unvalidated or Validated EDD - SNEDD format – 20070413.xls". When prompted, select Enable Macros.
4. Filter out samples in the EDD or SNEDD by Site and Media
5. Copy and Paste filtered records from the EDD or SNEDD into the worksheet "DATA" the correct formatting macro. Make certain that the column formats match.
6. Indicate validated records by updating the field "Valid_YN" on the "DATA" worksheet
7. Select Input Options
    a. Indicate validation status of the data, and enter header text for the data table.
8. Run Macro
    a. Select Tools / Macro / Macros.
    b. Select and run each macro in the following order: "CROSSTAB", "RAW_FORMATTING" and "DETECT_FORMATTING".
9. DO NOT SAVE DATA INTO THE MACRO!
10. Move formatted tables to a new workbook
    a. Highlight both worksheets "Raw" and "Detects".
    b. Right click one of the worksheets
    c. Select "Move or Copy"
    d. Select "Move to New Book", and click "OK"
11. Additional Table Formatting
    a. Ensure that Borders, Column Header Fonts, and Notes section at the bottom of the table look correct. (Note – CH2M HILL EDD will require the EIS to add the Notes Section by hand)
    b. Select File / Page Setup
        i. Page - Format tables to 11 x 17 paper, landscape orientation, 80 % size
        ii. Set Margins to: Top = 1.33, Left = .75, Bottom = .5, Bottom Footer = .35, Right = .5
        iii. Header/Footer
            1. Ensure appropriate header format. Ex. TO-45, NSN-CALF, Unvalidated Groundwater Raw Analytical Results, March 2006 LTM
            2. Ensure appropriate footer. Only page number should be displayed in the right.
        iv. Sheet – Ensure Gridlines is not selected.
12. Repeat for each Site and Media.